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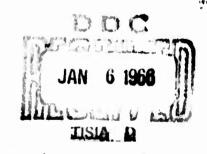
FEASIBILITY DEMONSTRATION OF PYROLYTIC GRAPHITE COATED NOZZLES

TECHNICAL REPORT NO. AFRPL-TR-65-57

March 1965

U.S. Air Force
Air Force Systems Command
Rocket Propulsion Laboratory
Edwards, California

Project No. 3059, Task No. 03



(Prepared under Contract No. AF 04(611)-9708 by the Atlantic Research Corporation, Alexandria, Virginia; J. D. Batchelor, E. F. Ford, and E. L. Olcott, Authors)

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FOREWORD

This report was prepared by Atlantic Research Corporation to document all work performed under Air Force Contract AF 04(611)-9708 from 2 February 1964 to 4 March 1965. This work was administered under the direction of the Air Force Rocket Propulsion Laboratory, Edwards, California. Lt. Robert Schoner was the Air Force Project Officer.

Dr. James D. Batchelor was responsible for the administrative and technical aspects of the program. The over-all program was carried out under Mr. Eugene L. Olcott, Director, Materials Division.

The secondary report number assigned to this report by the contractor is TR-PL-6839-01-0.

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(This abstract is Unclassified) ABSTRACT

Pyrolytic graphite coatings have been found to be highly erosion resistant under severe rocket nozzle conditions. The objective of this program was to demonstrate the feasibility of such coatings for nozzles up to 2.3-inch diameter in firings at 700 psi with a 6550°F propellant. This objective was accomplished.

Stress analyses and thermal analyses were carried out in support of the design, fabrication, and motor testing of nozzles of both 1.1-inch and 2.3-inch diameter. Thermal analysis indicated the potential of pyrolytic graphite coatings for lightweight nozzle designs. The results of the stress analyses for coated composites were correlated with experimental evidence of delamination cracking in the coatings. Critical stress levels were identified for both radial tension in the coating and axial tension in the substrate. The deposition process was improved to produce crack-free coatings 50-mil thick on conventional graphite substrates and 100-mil thick on a fibrous graphite substrate.

Nine motor firing tests were made and good performance was demonstrated with both subscale and full-scale nozzles. The presence of microscopically observed delamination cracks was found to destroy the integrity of pyrolytic graphite coatings during nozzle service. For coatings without major cracks coating integrity was achieved, however, and erosion rates of acceptable magnitude were measured for subscale nozzles for durations up to about 40 seconds. In full-scale nozzles satisfactory erosion rates were measured for firings of durations up to 60 seconds. Optimization of the coating to provide minimum erosion rates should result in still further performance improvements.

To test the restart capability of pyrolytic graphite coated nozzles, one subscale insert was re-fired successfully as an added task to the original program. This added task is described completely in the Addendum at the end of the report. Performance was completely normal and no cracking or delamination occurred. Pyrolytic graphite coated nozzles should be considered as good candidates for multiple firing cycle requirements.

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1.0 INTRODUCTION

Pyrolytic graphite is a unique form of graphite produced by vapor deposition of carbon from a carbonaceous source gas on a heated substrate. It exhibits excellent erosion resistance when used in uncooled nozzles for solid propellant rocket motors. Its high density, essentially zero permeability, and complete absence of a binder phase are believed to explain this exceptional serviceability. Subscale rocket motor tests of pyrolytic graphite coated nozzles of 1/2-inch throat diameter carried out previously at Atlantic Research Corporation have shown that consistently good performance can be achieved in nozzle service with propellants having flame temperatures from 5600 F to 6550 F. (Ref. 1) The higher the flame temperature and the motor operating pressure, the higher the erosion rate observed for pyrolytic graphite. To illustrate the high erosion resistance of pyrolytic graphite coatings, 1/2-inch diameter nozzles fired with 6550 F propellant have shown average erosion rates of the order of 0.5 mil/sec at 700 psi motor pressure. Limited tests with 1-inch diameter nozzles with a 6000 F propellant have also shown low erosion rates but the problems of scale-up have not been previously investigated sufficiently.

The lowest chemical reactivity can be achieved by using pyrolytic graphite as a coating so that the layer plane surfaces are exposed to the propellant combustion gas environment. However, to use the coating orientation, the difficulties and problems involved in maintaing coating integrity, which are common to all coating systems, must be accepted and solved. The anisotropic properties of pyrolytic graphite make the problem of maintaining coating integrity more severe than usual. Residual stresses are unavoidably built into the coating during the deposition process. The exposure of a pyrolytic graphite coating to the thermal shock conditions of nozzle service may also lead to coating compromise.

To circumvent the problems of maintaining coating integrity, many nozzle designers have resorted to the use of edge-oriented pyrolytic graphite in the form of stacked discs or washers. However, depending upon the heat sink capacity of the nozzle design and the severity of the service conditions, it has been found that edge-oriented pyrolytic graphite erodes up to several times faster than a good coating of pyrolytic graphite. A pyrolytic graphite coating is a good thermal insulator and coated nozzle inserts do not depend upon heat sink capacity to perform satisfactorily. Thus, a pyrolytic graphite coated nozzle can be designed for minimum volume and minimum weight. The potential exists, therefore, using pyrolytic graphite coatings, to prepare a nozzle of superior performance capabilities as well as minimum weight.

Previous work has been carried out to evaluate pyrolytic graphite coatings made by several suppliers (e.g., Ref. 2 and 3), but no definitive and systematic study of the feasibility of pyrolytic graphite coated nozzles of reasonable size and for severe motor operating conditions has been reported. Such a feasibility demonstration was the principal objective of this program.

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To investigate the feasibility and the performance capabilities of pyrolytic graphite coated nozzles for solid propellant rocket motors operating under severe conditions, the current programonsisted of the detailed analysis, fabrication and motor testing of a group of nozzles with an advanced propellant of 6550 F flame temperature. Five subscale nozzles of 1.1-inch nominal throat diameter for an 1100-lb thrust motor and four fullscale nozzles of 2.3-inch nominal throat diameter for a 4600-lb thrust motor were fabricated and motor tested. The preparation of the coated nozzle inserts, the fabrication of the test nozzles, and the rocket motor test of these units were all carried out at the facilities of the Atlantic Research Corporation. This report describes in detail the work carried out and the results achieved in this program.

2.0 SUMMARY AND CONCLUSTONS

The principal objective of this program was to demonstrate the feasibility of pyrolytic graphite coatings for uncooled nozzle service under severe operating conditions. Based on prior work in the field, it was apparent that a pyrolytic graphite coating represented a highly erosion resistant nozzle material. The utilization of this material, however, required a demonstration that coatings of suitable quality and integrity could be prepared and could perform successfully in rocket motor firings.

To accomplish this objective, both thermal and stress analyses were completed to aid in the design of the test nozzles and to aid in the understanding of their behavior during tests. Deposition studies were carried out to demonstrate that suitably coated inserts could be prepared for fabrication of test units for the motor firing program. Rocket motor tests of both subscale and fullscale nozzles were performed using a 6550 F flame temperature propellant at a nominal motor pressure of 700 psi. The motor firing program culminated in a demonstration of the ability of pyrolytic graphite coatings to withstand firings in excess of 60 seconds at high chamber pressure.

A number of interesting conclusions can be drawn from this program. The thermal analyses illustrated the unique insulating properties of pyrolytic graphite coating and provided design data for the successful fabrication and test of the nozzles built on this program. The stress analyses provided data on the residual stresses in composite bodies consisting of a pyrolytic graphite coating and the substrate upon which it was deposited. These data were not previously available in the literature. The effect of pyrolytic graphite coating thickness, substrate thickness, substrate radius, and substrate properties were illustrated by stress calculations. Based upon the stress calculations and the experimental evidence gathered in the deposition work, it was concluded that both the radial tensile stress in the pyrolytic graphite coating and the axial stress in the substrate material are critical in causing flaws in the coating.

In the deposition studies, it was determined that delamination cracking is the most common failure mode from residual stresses in pyrolytic graphite coated composites. For a given substrate material, delamination cracking occurred at similar coating thicknesses for both the subscale nozzles (1.1-inch diameter) and fullscale nozzles (2.3-inch diameter). This behavior indicates that critical stress factors may result both from the anisotropic nature of the pyrolytic graphite and from mismatch between the coating properties and the substrate properties. Control of delamination cracking was achieved both through deposition process control and through selection of the substrate material. Crack-free coatings of about 50 mil thickness were successfully prepared on conventional graphite substrates of both subscale and fullscale dimensions. Much thicker crack-free coatings were prepared on a low modulus, fibrous graphite substrate. Correlation of the calculated stress levels and the experimentally observed cracking was achieved.

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Nine motor firing tests (five subscale nozzles and four full scale nozzles) were made with a 6550 F propellant at 700 psi motor pressure. Excellent performance was demonstrated for both subscale nozzles (erosion rates from 0.55 to 0.64 mil/sec) and fullscale nozzles (erosion rates from 0.73 to 1.19 mil/sec). The higher erosion rates were measured for coatings on fibrous graphite substrates probably because of a greater tendency to form nodules in these coatings and the local flaws associated with nodules. The extent of coating deterioration and loss correlated with observed prefiring delamination cracks. Three coatings which contained known delamination flaws (two subscale and one fullscale insert) prior to firing were all destroyed during motor test. Of six coated inserts without observable major flaws, only one subscale unit suffered a complete loss of coating. Coating retention was demonstrated in the other five tests of inserts without cracks; four of these successful tests were consecutive in the latter portion of the program. Calculated stress values for both radial coating stress and axial substrate stress were correlated with the observed delamination cracks. The value of the pre-firing microscopic examination of the coating edges as a quality control procedure was clearly demonstrated by the program results. (C)

Post-firing microscopic examination of the coating on several inserts showed no deterioration of the remaining coating. This indicates that pyrolytic graphite coatings are a good candidate for restart nozzle applications. (U)

The feasibility of pyrolytic graphite coatings for use in uncooled nozzles for advanced solid propellant motors was demonstrated by the results of this program, but several areas are recommended for further study to optimize the coating performance and to advance the development of such nozzle assemblies. Stress analysis work should be extended to confirm the critical allowable stresses in coated composites and to optimize the control of these critical stresses. Deposition work should be aimed at the preparation of optimum quality coatings and the correlation of erosion resistance with microstructure. Repetitive motor firings of coated nozzles is recommended to demonstrate the indicated utility of pyrolytic graphite coatings for this type of duty cycle. Additional motor tests could provide experimental data for the correlations sought in further stress analysis and deposition process study. (U)

3.0 ANALYSIS AND DESIGN

A. GENERAL

It is important in the evaluation of nozzle materials that the proper design be used for each test unit. When the objectives is to evaluate the performance capability of the nozzle material, any short-coming of the design which compromises this performance is misleading and undesirable. To aid in the design of the test nozzles, both thermal analysis and stress calculations were carried out. Thermal analysis was carried out to determine the temperature history of the nozzle structure and to assure that thermal failures would not occur. The stress calculations carried out dealt with the residual stresses involved in the pyrolytic graphite coated insert. All of the test nozzles were constructed with heavy-weight test hardware to provide the maximum data on the nozzle inserts within the limited budget of the program. Design and optimization of a flight-weight nozzle were beyond the scope of this program.

Both the thermal analysis and the stress analysis calculations are discussed in some detail in this section because the results of these calculations indicate some of the unique properties of pyrolytic graphite coated nozzles. The thermal analysis indicates the potential for minimum weight nozzles offered by pryolytic graphite coatings. The stress analysis indicates the nature of the problems faced in preparing flaw-free coatings of desired dimension and in maintaining coating integrity during service.

B. THERMAL ANALYSIS

Pyrolytic graphite is an excellent thermal insulator in the direction normal to the deposition layer planes. When used as a coating this insulating property leads to a rapid rise in surface temperature, large temperature gradients across the coating thickness, and a significant reduction in the heat transmitted through the coating to the nozzle structure. The high surface temperature and large thermal gradients across the coating produce very severe service conditions for the pyrolytic graphite, but, if the coating can withstand these conditions, the reduction in heat transmission through the coating makes possible a very light-weight nozzle system. This characteristic is a distinct advantage for pyrolytic graphite coatings over conventional heat sink nozzles.

To predict the temperature history at several points in selected nozzle designs, a series of thermal analyses was carried out. Several cases were selected to provide a parametric study of the effect of coating thickness, substrate thickness, and insulating thickness for both the subscale and full-scale nozzles. The thermal analyses were performed with existing programs on a Burroughs 220 and an IBM 7090 computer. These programs utilize finite difference methods to calculate the transient radial conduction in axisymmetric cylindrical geometry and include the following features:

- (1) Convective heat input at the gas surface is based on Bartz correlation transfer coefficients and gas recovery temperature.
- (2) Radiative heat transfer at the gas surface is based on the particle cloud and surface emissivities and the gas free-stream temperature (i.e., no temperature lag in the condensed phase).
- (3) Temperature dependent thermal properties are used as needed for each material in the composite structure.
- (4) An adiabatic rear wall condition is assumed. Factors 1 and 4 are conventional assumptions generally made in all thermal analysis programs. The refinements of the program used in this study are the inclusion of temperature dependent thermal properties and the inclusion of radiative heat transfer between the nozzle surface and the propellant exhaust stream. The latter factor is quite important for long term firings when the surface temperature of the nozzle approaches a steady state value. If radiative heat transfer is ignored, significant error may exist in the calculated surface temperature. The use of a program for axisymmetric cylindrical geometry is also extremely desirable for nozzles of moderate diameter.

Calculations of the temperature histories were made for both subscale and fullscale nozzles at the nozzle throat location and at one upstream position in the nozzle inlet region. At the throat location, the composite structure consisted of pyrolytic graphite coating, graphite substrate, carbon back-up insulation, and steel structure. An edge-oriented pyrolytic graphite plate was used just upstream of the coated insert in the experimental nozzles to protect the leading edge of the coated section. Thus, the thermal analyses in the inlet region were made for a composite consisting of edge-oriented pyrolytic graphite, a baked carbon insulator, and the steel structure. In all calculations ATJ graphite was taken as the substrate material and baked carbon, which was used in all of the test nozzles, was taken as the insulation and back-up material. In each case, a standard thickness of 3/8 inch was used for the steel nozzle structure.

A total of fifteen cases were analyzed. The dimensions of each configuration selected for analysis are shown in Table I. The dimensions used for thermal analyses covered a range of dimensions anticipated to cover those used in actual experimental nozzle designs. The data calculated for each case are summarized in tabular form in Appendix A. The reader may refer to these data for any detailed comparisons desired. Selected data are discussed and compared in the following paragraphs to illustrate key features of pyrolytic graphite coated nozzles.

Several interesting facts may be noted by examining the predicted temperature histories. First, the very strong effect of the insulating nature of the pyrolytic graphite coating is apparent from the rate at which heating of the nozzle occurs at the throat region compared to a nozzle without the

Table I. Dimensions of Nozzles Selected for Thermal Analysis.

A. Throat location: 1.100" throat diameter; 4.750" outside diameter; steel thickness 3/8"

Case No. a	Pyrolytic Graphite Coating Thickness (mil)	Graphite Thickness (inch)	Graphite/Carbon Interface Diameter (inch)	Carbon Thickness (inch)
A-1	0	0.700	2.500	0.75
A-2	0	0.950	3.000	0.50
A-3	30	0.670	2.500	0.75
A-4	30	0.920	3.000	0.50
4-5	60	0.640	2.500	0.75
A-6	60	0.890	3.000	0.50

B. Throat location: 2.300" throat diameter; 6.250" outside diameter; steel thickness 3/8"

Casa No.	Pyrolytic Graphite Coating Thickness (mil)	Graphite Thickness (inch)	Graphite/Carbon Interface Diameter (inch)	Carbon Thickness (inch)
3-1	0	0.850	4.000	0.75
B-2	0	1.100	4.500	0.50
8-3	45	0.805	4.000	0.75
3-4	45	1.055	4.500	0.50
B-7	45	0.805	4.000	1.125 ^b

C. Inlet location: 1.840" diameter (1.100" throat); steel thickness 3/8"

	Pyrolytic Graphite	Carbon	Outside
Case No.	Plate Web Thickness (inch)	Thickness (inch)	Diemeter (inch)
C-1	0.830	0.50	5.250
C-2	0.830	0.875	6,000

D. Inlet Location: 3.540" diameter (2.300" throat); steel thickness 3/8"

	Pyrolytic Graphite Plate Web Thickness	Carbon Thickness	Outside Diameter
Case No.	(inch)	(inch)	(inch)
D-1	0.855	0.625	7.250
D-2	0.855	1.000	8.000

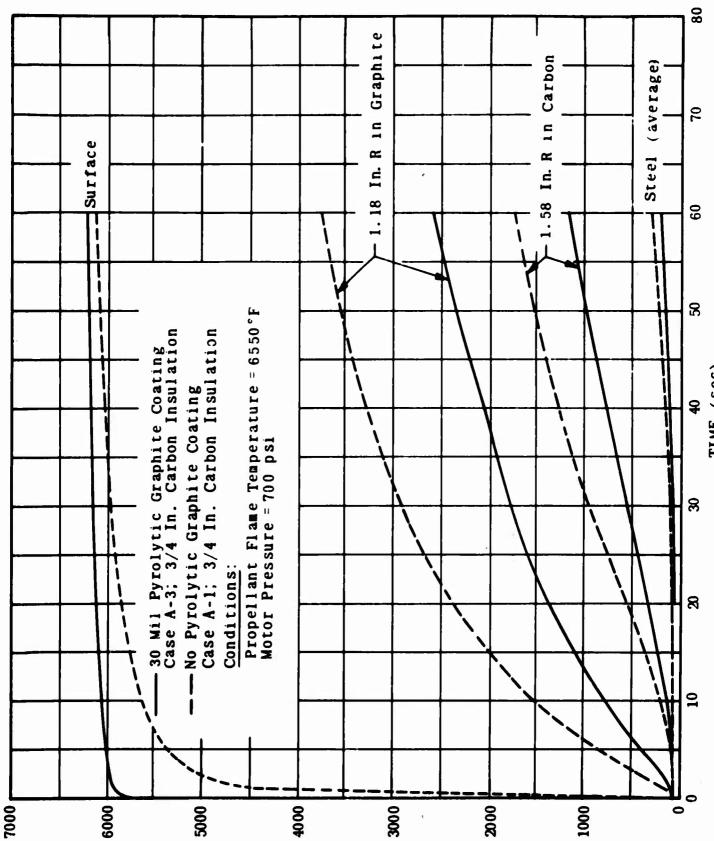
a. Results of these cases are found in Appendix A.

b. Outside diameter for case B-7 - 7.000"

pyrolytic graphite coating. (Figure 1) This insulating property of the coating leads to the predicted very rapid rise in surface temperature. Just how rapidly this rise occurs can be seen in Figure 2 in which the surface temperature of the throat of a subscale nozzle with only 30 mils of pyrolytic graphite coating is shown with a greatly expanded time scale. It can also be seen from these temperature plots that practical thicknesses of baked carbon in the range from 1/2 to 1 inch provide adequate insulation between the nozzle throat insert and the steel housing for all the firing conditions planned in this program (Figures 1 and 4).

The temperature histories predicted in the inlet region of both the subscale nozzle and the fullscale nozzle are shown in Figures 3 and 5. A substantial temperature rise can be expected from the heat sink, edge-oriented pyrolytic graphite plate, but this need be no cause for concern for heavy-weight nozzle test hardware. The edge-oriented pyrolytic graphite plate is included for the primary purpose of providing protection for the leading edge of the coated insert section. In previous work, excessive erosion upstream of the coated insert has led to unduly severe exposure conditions of the coating. In practical nozzle designs, no greater weight of the pyrolytic graphite plate material would be desirable beyond that needed to provide protection for the upstream edge of the coating. To optimize a light-weight nozzle design, another method of controlling the entrance contour, such as the use of a second pyrolytic graphite coated segment, might be found more favorable.

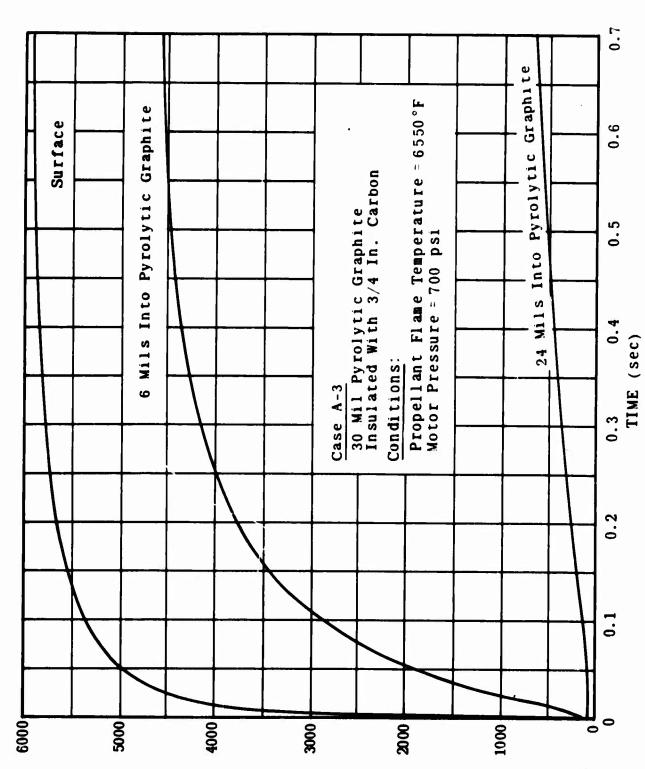
The dimensions of the nozzles which were motor tested were comparable with cases subjected to thermal analysis. The thermal calculations indicated that no problems would be encountered with over heating of the nozzle structures. These predictions were borne out in the motor test firing program.



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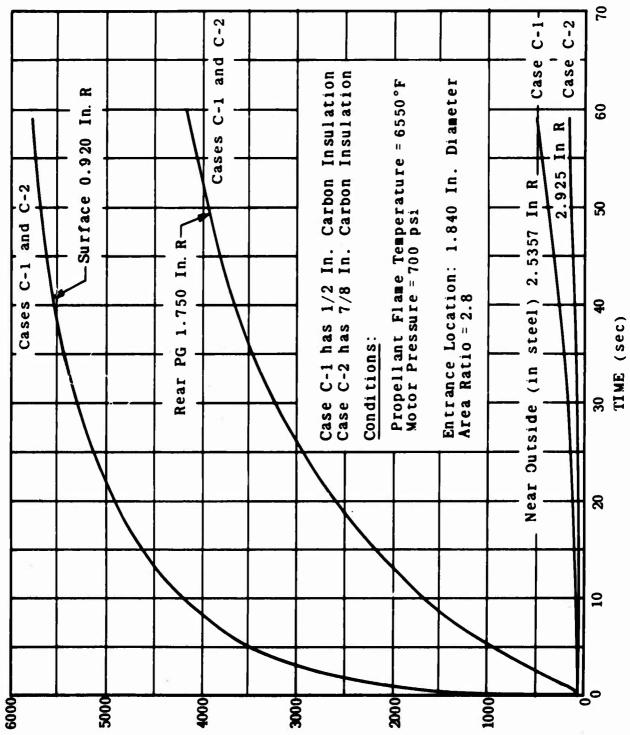
CALCULATED TEMPERATURE AT THROAT (*F)

Figure 2. Initial Temperature Rise in Pyrolytic Graphite Coating on Subscale Nozzle.

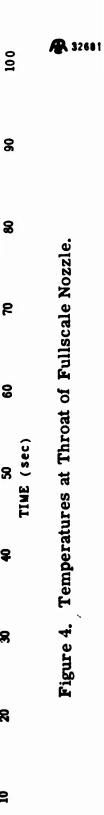


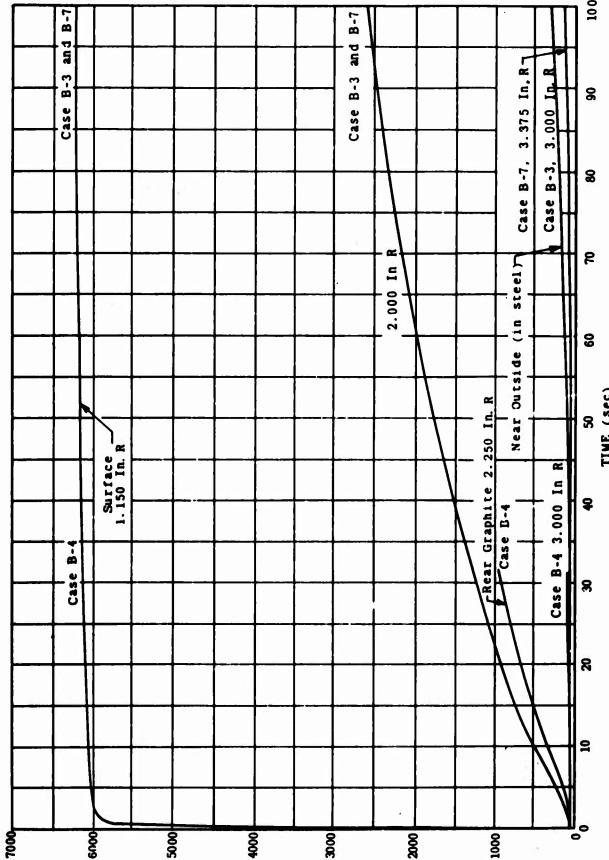
CALCULATED TEMPERATURE AT THROAT (°F)

. Figure 3. Temperature in Entrance Section of Subscale Nozzle.

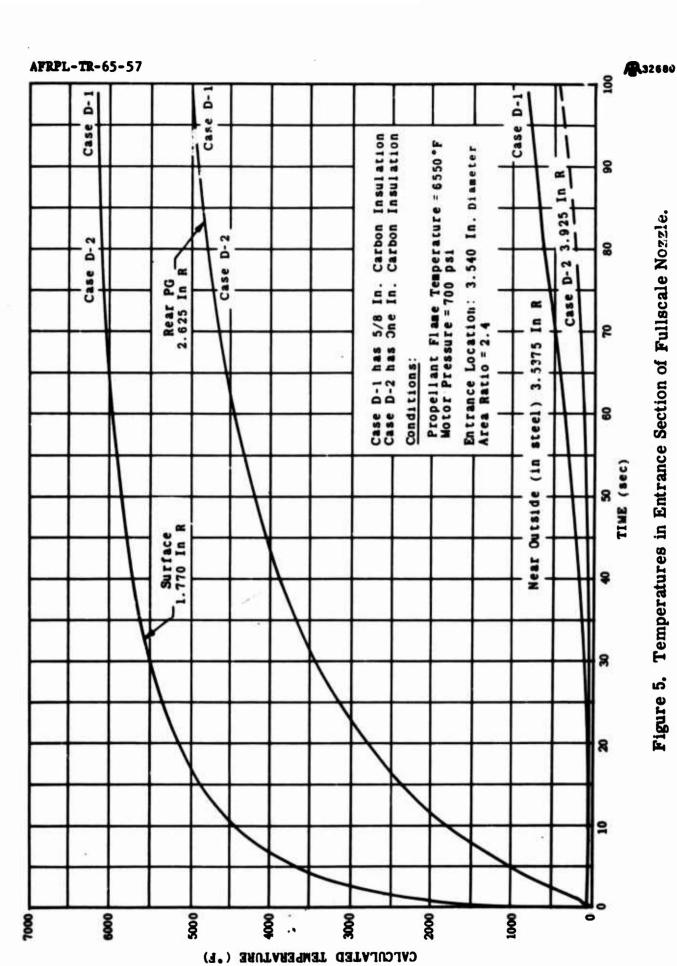


CALCULATED TEMPERATURE ("F)





CALCULATED TEMPERATURE AT THROAT (*F)



C. STRESS ANALYSIS

In the design of any nozzle, various stress factors must be considered. Stresses exist in the throat insert, in the back-up insulation, and in the nozzle structure. In a flight-weight nozzle design, the stress levels in the nozzle structure and attachments assume equal importance with the stresses in the internal components of the nozzle. This is true because each component must be designed to save weight. When heavy-weight nozzles are designed for test purposes, however, the structural stress problems may be minimized by conservative design. The conservative design for the test hardware used on this program contributed to the satisfactory performance experienced with the test nozzles to yield maximum data on the coating performance.

Stress factors in the nozzle throat insert material under evaluation cannot be disposed of easily. These stress factors and their effect upon the nozzle material are often central to the performance capabilities of the material. Residual stresses may be incorporated in the nozzle insert material during its manufacture. Additional stresses or modification of the residual stress pattern may be caused during a rocket firing both by the severe thermal environment and the mechanical forces acting upon the nozzle. Often one of these stress types is critical for the application of 2 given nozzle material. Such was the case in this program as indicated in the following discussion.

Pyrolytic graphite is a highly anistropic material. Because the material consists of a stacked layer plane structure, the thermal and mechanical properties are very different in the a-b direction (parallel to basal planes) in the c- direction (perpendicular to the basal planes). The elastic constants of the material also differ in the a-b and c directions. A material which exhibits this type of anisotropy, in which the properties in two directions (a-b) are equivalent but differ from those in the third direction (c), is properly referred to as transversely isotropic. It is known that a transversely isotropic material is prone to develop residual stresses when its temperature is changed. A change in the temperature of the material is a sufficient condition to induce stresses; it is not necessary for a temperature gradient to exist in the material as is the case for an isotropic material. In the case of a flat piece of pyrolytic graphite, the stresses induced during cooling from the deposition temperature lead to warping or distortion of the surface. However, in closed shells of pyrolytic graphite, the geometrical restraint offered by the closed shape locks in residual stresses.

The residual stresses inherent in pyrolytic graphite coatings represent the single most critical stress factor in pyrolytic graphite nozzles. Although the general nature of the residual stresses in pyrolytic graphite are known, little definitive information has been published on the magnitude of these stresses especially for the composite consisting of a pyrolytic coating and its graphite substrate. An approximate analysis of the stresses in free-standing spherical and cylindrical shells of pyrolytic

graphite was reported in Reference 4. In both references 4 and 5, the local effects of nodules and surface roughness were explored for free-standing geometry. Reference 6 contains, in addition to the work published in References 4 and 5, a variety of brief and exploratory analyses of many of the different stress problems such as growth, thermal gradients, surface roughness, etc., discovered in the course of the fabrication of pyrolytic graphite shapes. Calculations are made only for free-standing shells although the effects of mandrel restraints are suggested. Perhaps most important in Reference 6 is the exploration of methods of measuring residual stresses by strain gage techniques. It appears that with sufficient care and extensive effort residual stresses can be measured, but such measurements require destruction of the pyrolytic graphite section. The data in the literature is of general interest, but it does not relate the stresses in coated composite structures to the geometry and properties in the way required to guide a study of pyrolytic graphite coated nozzles.

The principal mode of failure of coated inserts resulting from residual stress consists of delamination cracks which are parallel to the substrate and coating free surface. Such cracks may form at the substrate coating interface or completely within the coating and once initiated generally propogate around the nozzle circumference. Radial tensile stresses in the coating are normal to such cracks and could be responsible for delamination. Shear stresses resulting from axial strusses in short coated pieces could also produce interlaminar failure. The axial curvature of the nozzle would lead to an opening of an interlaminar crack following a shear stress failure. Microscopic examination of the polished edge of the coating readily reveals delamination cracks; the prediction and control of such flaws was the main objective of the stress analyses. Short interlaminar cracks are often found in the region of growth modules in local areas of otherwise sound coatings. These defects, which are referred to as local flaws because they do not form a continuous crack pattern, would likely be less destructive coating integrity.

To define the problems inherent in the as-deposited coatings under study in this program, a series of stress analyses was carried out. Calculations were made on the effect of geometrical variables on the residual stress in free-standing pyrolytic graphite shells. Calculations were also made for the effect of geometry and substrate properties on the stresses in composites consisting of a pyrolytic graphite coating and its substrate. The method used in these calculations and the results of each type of calculation are discussed in the following sections.

Method of Analysis

In a previous program at Atlantic Research (Reference 1), the basic stress-strain relations for pyrolytic graphite were derived. These relationships for a transversely isotropic material are much more complicated than for the case of an isotropic material. The stress-strain relationships involve seven elastic constants rather than three for an isotropic case. However, only five of these elastic constants are independent.

In our prior work, these stress-strain relationships were used to derive the differential equations relating the stresses and the deflections for a cylinder of pyrolytic graphite. These equations were the starting point for the calculations of stresses in pyrolytic graphite shapes which can be described in cylindrical coordinates.

To carry out the desired stress analyses on this program, computer programs were written to solve the basic equations on a Burroughs 220 computer. The first program prepared dealt with the stresses in a homogeneous free-standing cylinder of pyrolytic graphite. This program was used to calculate the stresses in long cylinders in which the edge effects were not involved. The input data required for this program were the thermal and elastic properties of the pyrolytic graphite and the temperature at which the material was in a stress-free condition. The output of the program consisted of the principal stresses at any other temperature.

A second computer program was also prepared in which the pyrolytic graphite coating equations were coupled with those which apply to a cylindrical substrate material. This program could be used to calculate the stresses in both the coating and the substrate for composites in which the coating is on either the inside or the outside of the substrate cylinder. This program was particularly important to our study of pyrolytic graphite coated inserts. The interaction of the coating and the substrate cannot be ignored if pyrolytic graphite is to be used on an as-deposited coating basis. No serious study of the stresses in as-deposited composites was available from previous work so that this portion of our stress analysis was critical in the guiding of our deposition studies and the interpretation of the results of this deposition work.

2. Free Standing Pyrolytic Graphite Shells

As indicated above, the anisotropic nature of pyrolytic graphite leads to residual stresses in any shell when the temperature of the material is changed. Although these stresses cannot be eliminated or avoided, an identification of the nature and magnitude of these residual stresses is of value in the design of a nozzle.

The stresses in long cylinders of free-standing pyrolytic graphite were calculated using anisotropic elastic and thermal expansion properties for the pyrolytic graphite. The stresses considered in each case are those created by cooling the material, assumed stress free at the deposition temperature of 2000°C, to room temperature. The thickness of the pyrolytic graphite shell and its diameter were varied over a range typical of those values of interest for the nossles in this program. The maximum tensile stresses calculated in each of the eight cases considered are listed in Table II. A complete description of the stresses calculated and the input data used in this series of calculations are contained in Appendix B. Compressive stresses are not listed in Table II since failures caused by such stresses are rarely observed and were certainly not involved in the cracking found experimentally in this program. It must be remembered that the stresses calculated for free-standing pyrolytic graphite shells are

Table II. Summary of Residual Stresses in As-Deposited Free-Standing Cylinders of Pyrolytic Graphite.

Case No.	Inside Dia.	Coating Thickness (mil)	t/r ₁	Radial ^C (psi)	Max. Tens Hoop (psi)	Max. Tensile Stresses Hoop (psi) (psi)
1	1.120	30	0.0536	99	5,120.	744.
2	1.120	20	0.0894	176	8,470	1,166
٣	1.120	80	0.143	427	13,400	1,660
4	2.300	30	0.0261	16	2,500	832
s	2 .300	20	0.0435	77	4,160	919
9	2.300	80	9690.0	109	6,620	936
) £	0.200	006	9.0000	57,800	206,000	19,100
&	2.240	100	0.0894	176	8,470	1,160

. Complete computer data may be found in Appendix B.

o. Ratio of coating thickness to inside radius.

c. Maximum radial tension occurs at mid-plane position.

Maximum hoop tension occurs at inside surface; comparable compression exists at outside surface.

Maximum axial tension occurs at outside surface; comparable compression exists at the inside surface.

Extremely thick walled cylinder to prove that stresses to continue to increase as thickness to ratio increases. not those which exist in as-deposited coated composites. However, these residual stresses which exist in pyrolytic graphite shells without regard for restraints from the substrate are of interest as background for the study of coated composites.

Several interesting conclusions can be drawn from the data in Table II. First, the radial tensile stresses, which might explain the delamination cracks which are most common in pyrolytic graphite, are not excessive for dimensions which are realistic for our nozzle study. An allowable radial tensile stress of about 1000 psi (across plane direction) is a reasonable value for pyrolytic graphite. Case 7 which was included to show the effect of a very thick shell of small inside diameter indicates that extremely high stresses would characterize such an unfavorable configuration.

In addition to the stress levels, the effect of the thickness (t) to inside radius (r_i) ratio on the stresses is of interest. Cases 2 and 8 indicate that geometric similarity leads to equal stresses in thin shells. The stress in each coordinate direction increases with increasing t/r_i ratio but not in the same manner. Over the range of t/r_i ratios typical of the nozzles for this program, the following observations can be made:

- (1) the hoop stress increases essentially in proportion to the increase in $t/r_{\rm c}$,
- (2) the axial stress increases with increased $t/r_{\rm r}$ but at a rate of less than proportional to the increase in $t/r_{\rm r}$, and
- (3) the radial stress increases with increased $t/r_{\rm r}$ but at a rate greater than proportional to the increase in $t/r_{\rm r}$.

3. Coated Composites

The nozzles under study in this program were all coated composites consisting of a coating of pyrolytic graphite as-deposited on the inner surface of a commercial graphite substrate. Since the pyrolytic graphite was to be used as an as-deposited coating, the stresses calculated for the composite are of greater interest than those calculated for free-standing shells. In any pyrolytic graphite coated body some mismatch of thermal expansion coefficients exists between the coating and the substrate in one or more directions. The elastic properties of the substrate graphite also generally differ from those of pyrolytic graphite. Each of these differences, in addition to the anisotropic nature of the pyrolytic graphite, contributes to the residual stress in a coated part. Again, these stresses cannot generally be eliminated but their identification is important. Effective control of the magnitude of these stresses is possible by the proper selection of geometrical factors and the properties of the substrate material.

Three series of stress calculations were made for coated composites consisting of a long cylindrical configuration. In a long cylinder, only

principal stresses exist and no shear stresses are developed. The selection of this configuration was necessary to avoid the mathematical complication of edge discontinuities which are involved in short cylinders. Thus, these calculations represent a partial solution and simplification of the stress pattern in actual coated inserts. However, valuable insight was gained into the nature of the stresses and the relation of these stresses to the geometry and materials selected for coated inserts.

a. Coatings on Inner Surface of Cylinder

In the first series of calculations, the stresses were computed for composites consisting of a pyrolytic graphite coating on the inner surface of a cylindrical substrate. Twenty-four cases were analyzed to determine the stress levels for all combinations of the following variables:

- (1) pyrolytic graphite thickness 30, 50 and 80 mils,
- (2) substrate thickness 0.40 and 0.70 inch,
- (3) substrate type molded and extruded graphite,
- (4) inside diameter 1.120 and 2.300 inch.

Table III contains a summary of the results of these calculations. Only the maximum principal tensile stresses are listed although compressive stresses of similar magnitudes are present. No failures which appear to be the direct consequence of compressive stresses have been noted in the experimental work. A complete presentation of the results of these stress calculations is included in Appendix C. The reader who is interested in further comparisons or study of these results is referred to this tabulation.

Examination of Table III illustrates a number of interesting factors. For a given type and size of substrate the radial tensile stress increases with coating thickness. If r substrates of the same size, the radial tensile stresses for an extradad graphite substrate exceed those for a molded graphite. The magnitude of these radial tensile stresses are particularly important because they could explain delamination cracks. For a number of the configurations listed in Table III the maximum hoop tensile stress in the coating becomes quite large. Only in the case of the thicker coatings on the smaller substrate, however, does this hoop stress approach the value generally reported for the tensile strength of pyrolytic graphite in the a-b direction.

The axial stresses in the coating are entirely compressive. The tensile stresses in the axial direction in the substrate, however, are indicative of the forces created by the mismatch of the coating and the substrate in the axial dimension. The axial tensile stresses in the substrate increase with coating thickness and decrease with substrate thickness. For the cases selected, the axial stresses are somewhat larger in the large diameter substrate than in the smaller diameter substrate. The axial stress

Table III. Summary of Residual Stresses in Cylindrical Composites with Pyrolytic Graphite Coatings on the Inside.

	Substrate	laside.	Substrate	Coetie	in in	Continue		1	September	
Case No.	Graphite	Gas Io. Tree (tack)	Hell Thickness (fach)	factors.		13		ests de	ू (देव इंड	
-	Molded	1.120	0.400	8	į	1	į	į	639.	1,150.
~				8	32.2	3,370.			701.	1,763.
•		•		8	277.	10,170.	'n		497.	2,486.
•			0.700	8	None	Hone			*	592.
S				8	38.9	3,670.	1	*	457.	£9.
•				2	305.	10,610.	x ;	*	231.	1,418.
7	8	2.300	0.400	8	Kose	Hone		•	724.	1,296.
•	•	:		8	Kos	Hose	E		1,013.	1,979.
•		*	=	8	0.9	557.	2	2	1,243.	2,800
9	•	2	0.700	8	Fos.	Mos	*		469.	716.
11		:		8	N S				159	1,136.
12			=	2	1.1	603.	:	į	785.	1,6%.
13	Ertruded	1.120	0.400	8	13.1	2,200.	:	128.	1,925.	1,193.
2		2	: :	8	137.	7,240.		147.	1,730.	1,499.
21		*	2	8	419 .	14,020.	X	153.	1,200.	1,835.
91			0.700	8	57.6	4,630.	*	397.	2,686.	1,259.
17		2		8	238.	9,580.		424.	2,400.	1,424.
18		r	1	8	659.	16,320.	\$	3 6	1,807.	1,627.
61		2.300	0.400	8	Kone	Hone	ŧ	10.	1,427.	3
20		8		8	Kone	e de	E	'n	1,571	1,351.
21		2;	=	8	24.0	3,000			1,593	1,783.
2	2	1	0.700	8	None	208		Ŗ	1,757.	893.
23		:	=	8	Kons	162.	*	.5	1,000.	1,111.
77	*	:		2	43.7	4,040.		į	1,740	1,396.
. Comple	ste computer	date may be for	and in Appendix C.							

is important in coated composites because in actual nozzle inserts edge effects come into play which yield secondary stresses in the composite. The transfer of axial stresses across the interface between the coating and the substrate in actual sections of finite length creates shear stresses. It is likely that these shear stresses, which are inter-laminar in nature for the coating, are one cause of the delamination flaws. The analysis of these shear stresses was not carried out in this program as indicated above because of the difficulty of such analysis. However, it is estimated that the magnitude of the shear stresses is related directly to the magnitude of the axial tension in the substrate.

Finally, examination of the hoop tensile stresses in the substrate indicates another potential problem area. In the cases involving the molded graphite, the hoop tension in the substrate appears safe since the tensile strength of molded graphite is of the order of 3000 spi. However, the tensile strength of extruded graphite, which may range from 1200 to 2000 psi, may not be sufficient to withstand the hoop or axial stresses. In fact, in the experimental deposition work, several instances of hoop stress failure occurred in extruded graphite substrates.

b. Coatings on Outer Surface of Cylinder

In the first series of calculations described above, the nozzle shape was idealized as a composite cylinder with the pyrolytic graphite coating on the inner surface. The cylinder was selected such that the circular cross-section was typical of a given nozzle throat plane. Thus, these previous calculations served to illustrate the effect of nozzle throat radius on coating stresses. To illustrate the effect of axial nozzle curvature on the stresses in the coated insert section, calculations were made for a composite cylinder with the coating on the outside with the cylinder radius equal to the radius of curvature of typical nozzles. Figure 6 shows pictorially the rationale behind this selection of geometry. The surface of an actual nozzle insert represents only a portion of a toroidal surface and thus does not consist of a closed surface along the direction of the radius of curvature. Nevertheless, it was felt that calculations for this outside coating geometry would help to indicate the effects of nozzle radius of curvature to supplement the calculation of the effects of nozzle throat diameter.

Twelve cases were calculated for coatings on the outside of a cylinder to demonstrate the effect of two radii of curvature, two thicknesses of a molded graphite substrate, and three pyrolytic graphite coating thicknesses. The maximum tensile stresses in each principal direction in both the coating and the substrate are listed in Table IV. Again only the maximum tensile stresses are listed although compressive stresses also exist. A complete tabulation of the stresses in each direction is included for each case in Appendix D. Examination of Table IV indicates that increasing the substrate thickness increases the radial tensile stress, but decreases the axial stress in the substrate. Increasing the radius of the substrate (i.e., the radius of curvature of the nozzle) decreases the stress levels. It was

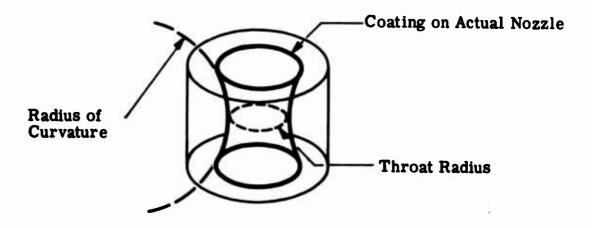


Figure 6. Pictorial Diagram Showing Parameters Related to Nozzle Slope.

Table IV. Summary of Residual Stresses in Cylindrical Composite with As-Deposited Pyrolytic Graphite Coatings on the Outside. a

1				Maximum Tensile Stresses in Coating			Maximum Tensile Stresses in Substrate			
Case No. a		Coating Thickness (mil)	Radial (psi)	Hoop (psi)	Axial (psi)	Radial (psi)		Hoop (psi)	Axial (psi)	
	A.	Substrate	radius =	1.600";	substrate	thickness	=	0.100"		
1		30	277	ъ	ъ	277		4440	2710	
2		50	384			384		6210	3860	
3		80	500			500		7900	5080	
	B.	Substrate	radius =	1.600";	substrate	thickness	=	0.400"		
4		30	382			382		1900	1050	
5		50	610			610		2710	1580	
6		80	925			925		3840	2290	
	C.	Substrate	radius =	3.200";	substrate	thickness	=	0.100"		
7		30	134			134	10	4300	2650	
8		50	182			182	•	5820	3760	
9		80	232			232		7370	4890	
	D.	Substrate	radius =	3.200";	substrate	thickness	=	0.400"		
10		30	188			188		1680	910	
11		50	294			294		2470	1400	
12		80	431			431		3510	2070	

a Complete computer data may be found in Appendix D.

b. Where no value is listed, stresses are entirely compressive.

not feasible because of design limitations and deposition problems, to investigate experimentally the apparent improvement gained by going to a larger radius of curvature. Furthermore, increasing the radius of curvature generally produces a rather severe penalty in a light-weight nozzle by increasing the length of the nozzle.

Several other interesting stress patterns are apparent from examining Table IV. The only tensile stresses in the coating are those in the radial direction. In the substrate, the radial tensile stresses are equal to those in the coating but both the hoop and axial stresses are also tensile. Both the hoop and axial stresses are quite high for the thinner substrates. Increasing the substrate thickness, of course, decreases both of these stress levels. As would be expected, increasing the coating thickness, other factors being equal, increases all the stress levels.

c. Effect of Substrate Properties

In the discussion above of the source of the stresses in pyrolytic graphite coated inserts, it was pointed out that residual stresses could not be eliminated. However, it is apparent that the properties of the substrate graphite will have a significant effect on the stress levels in both the coating and the substrate portion of a coated composite. In an effort to identify preferred substrate graphites, a series of calculations was made for a molded graphite (National Carbon ATJ), an anisotropic, high-density graphite (National Carbon ZTA), a low-modulus, fibrous graphite (National Carbon PT-0114), and an extruded graphite (Speer 580). Calculations of the stress levels in both substrate and coating layers were made for composite cylinders with the coating on the inside using two diameters, equivalent to the two nozzle sizes under study, and three coating thicknesses for each of the four substrate materials. The complete results of these calculations are tabulated in Appendix E. The input data used for these calculations are also tabulated with the results. The thermal expansion values used were somewhat different from those used in the earlier calculations. The data which were used in this last series of analyses were taken from the latest and most complete sources available in the literature. Therefore, for correlation of calculated stresses with experimental data these results are most reliable.

The results of this series of calculations are summarized in Table V. Various effects of the substrate properties are apparent from examination of this table. The stresses calculated for the molded graphite (ATJ) appear moderate except for two factors. As the coating thickness increases in relation to the diameter (e.g., Case 3), the radial and hoop stresses in the coating rise quite rapidly. This appears to be evidence of the internal anisotropy of the pyrolytic graphite which is not greatly modified by the ATJ substrate. Second, the axial stress in the substrate for 50 and 80 mil coatings is higher than for the other substrates compared in Table V. The shear stresses

Table V. Effect of Substrate Properties on Stresses in Composite Cylinders.

			Maximum Tensile Stresses	nsile St	resses	Maximum Tensile Stresses	nsile Str	e 8 8 e 8
	Inside	Coatino	III	in Coating	ļ	In	in Substrace	
Case No.	Diameter (inch)	Thickness (mil)	Radial (psi)	Hoop (psi)	Axial (psi)	Radial (psi)	Hoop (ps1)	Axial (psi)
A.	Molded substrate	rate (ATJ) 0.700" thick	thick					
1	1.120	30	.:	638	Þ	Ą	322	267
7	1.120	50	93.	6010			262	006
m	1.120	80	425.	13,200		18		1310
4	2.300	30					380	089
S	2.300	20					505	1,070
•	2.300	80	18	2,670			565	1,560
æ	High Density sub		(anisotropy	same or	trate (ZTA) (anisotropy same orientation as PG); 0.700" thick	3); 0.700" €	hick	
7	1.120	30	.	Ą	1,680		3,770	Д
•	1.120	20					5,360	23
6	1.120	80					6,880	154
10	2.300	30			2,220		3,040	
11	2.300	20			655		4,580	
12	2.300	80					6,360	124

Complete computer data may be found in Appendix E.

b Where no value is listed, stresses are entirely compressive.

Table V. (cont'd)

			Maximum T	Maximum Tensile Stresses in Coating	6 8 6 8	Maximum 1n	Maximum Tensile Strasses in Substrate	ET :3 8 6 8
Case No.	Inside Diameter 1 (Inch)	Coating Thickness (mil)	Radial (psi)	Hoop (psi)	Axial (psi)	Radiel (psi)	Hoop (psi)	Axial (psi)
ပ်	Molded Fibrous su	substrate (PT-0114); 0.700" thick	0114); 0.7	00" thick				
13	1.120	30	111	6,830	٩	74	Þ	189
14	1.120	50	27.1	10,800		154		256
15	1.120	80	809	16,500		280		310
72	2.300	30	22	2,980		6		220
ټ.	2.300	50	19	5,050		28		297
18	2.300	80	151	8,040		99		363
Ö.	Extruded substrate (Speer 580); 0.700" thick	e (Speer 580)	. 00.100.	thick				
19	1.120	20	873	20,300	712	873	1080	896
20	1.120	80	1520	27,200	2200	1490		842
21	1.120	120	2500	35,700	4040	2290		712
22	2,300	50	243	10,400	17	268	161	516
23	2.300	80	440	14,100	149	439	287	909
54	2.300	120	750	18,700	1730	722		457

Complete computer data may be found in Appendix E.

Where no value is listed, stresses are entirely compressive.

" ich result from the axial force in actual insert sections of short length can explain the observed delamination flaws.

With the high density graphite (ZTA), the calculations show that the radial tension is eliminated and the axial tension in the substrate is greatly reduced or eliminated for all cases. Two major problems can be identified, however. First, the substrate hoop tension exceeds the material strength for all configurations. Consistent with this, the only coating deposited on ZTA graphite led to a hoop tension failure in the substrate. Second, the orientation of the substrate anisotropy cannot actually match the coating anisotropy orientation as assumed in the calculation since the ZTA is a molded graphite. The experimental substrate was cut with its axis across the molded billet so that the hoop and radial properties are alternately with-grain and across-grain at 90° intervals of rotation. Matching of the axial direction of the substrate with the with-grain direction of the ZTA, to provide the reduction in axial stresses, was achieved.

Perhaps the most significant results in Table V, at least for support of the firing tests in the current program, are those for the fibrous graphite (PT-0114). Each stress calculated, with the possible exception of the thickest coating on the subscale substrate appears to be of a tolerable magnitude. Experimental evidence confirms these predictions since coatings 51 mils thick on the subscale substrate and 100 mils thick on the fullscale substrate were deposited without stress failure. The calculated axial tension in the substrate is of the same order as the published cross-grain strength of the material (Ref. 11), but no evidence of substrate delamination was noted. It is quite possible that some permanent deformation of this type of substrate is possible without the appearance of discrete cracks. Although the low grade properties of fibrous graphite do not recommend it on first examination as the most desirable substrate, the use of this type of substrate did provide a useful advance in the technology of coated inserts for fullscale tests in this program. Further work will be required to determine whether another substrate selection would be optimum or whether the fibrous material does represent a preferred approach.

The extruded graphite (Speer 580) was selected because it had better-than-average strength properties for an extruded grade and an unusually low thermal expansion in the with-grain (axial) direction. The improved match in expansion in the axial direction leads to reduced axial tension in the substrate as seen in Table V. Both the radial and hoop stresses in the coating on this extruded graphite were calculated to be higher than for the molded (ATJ) substrate. Assuming a dual criterion for coating integrity, namely, low radial stress in the coating and low axial stress in the substrate, the calculations predict qualitatively the observed results for coatings deposited on Speer 580 graphite. Coatings up to 47 mils in thickness were uncracked, but at 64-mil thickness delamination cracks were noted. It should be noted that because of the interest in thicker coatings for the final fullscale motor firing,

the coating thickness considered for the calculations in Table V for the Speer 580 was increased to 120 mils. A more detailed comparison of the calculated stresses in coated inserts with experimental observation of delamination cracks is made after the description of the deposition studies in the next section of this report.

4.0 DEPOSITION STUDIES

A. GENERAL

Rocket motor firing tests were the basis of the feasibility demonstration of pyrolytic graphite coated nozzles. However, no motor firings could be achieved without the prior preparation of suitable inserts. All of the inserts tested in this program were fabricated in the laboratories of Atlantic Research Corporation. The integration of the deposition studies and the motor test evaluation work was a very distinct advantage in this program. Deposition work was carried forward until inserts suitable for testing were produced. Following the test in a motor firing of a given insert, it was possible to return immediately to the deposition study in order to prepare further and improved coated inserts. In this way, the maximum feedback of information from the evaluation phase was available to guide the deposition work.

The equipment and procedures used in the deposition of pyrolytic graphite coatings was fully described in Reference 1. All of the coatings were deposited at a substrate temperature of 2000°C with methane as the carbonaceous source gas. The density of coatings prepared under these conditions is typically 2.17 gm/cu cm. X-ray diffraction indicates typical values as follows:

d-spacing = 3.45 Å

crystallite size: Lc = 75A°; La = 120Å

The deposition conditions which were varied to control the nature and thickness of the coatings included the flow geometry, the flow rates, and the deposition time. Deposition work was carried out to prepare subscale inserts of nominal 1.1-inch throat diameter and fullscale inserts of nominal 2.3-inch diameter. The results of the deposition study in each size range are discussed separately.

B. SUBSCALE INSERTS

The initial deposition work was carried out with subscale inserts. Since the basic design of the test nozzle utilized an insert segment coated with pyrolytic graphite which was only long enough to provide the required dimensional stability at the throat of the nozzle, an excellent opportunity existed to study the quality of the coated insert at both the upstream and downstream edges of the insert. The examination of the microstructure and quality of the coating was carried out by microscopic examination of the polished cross-section. This examination is indispensible if the performance of the nozzle is to be related to the pre-firing condition.

The radius of curvature of the subscale insert was chosen to be three times the nominal throat radius. The length of the coated insert was

selected as 1.22 inches so that the inlet joint fell at an area ratio of 2 relative to the throat and the outlet joint occurred at the 15° tangent point to match the expansion cone half angle. Several coated inserts were prepared on molded graphite substrates of this configuration prior to the first motor test. In each insert, significant delamination cracking was found at the exit end of the coating during microscopic examination. The cracks were always in the coating, never at the substrate surface. These cracks often originated during the machining of the insert piece from the coated substrate tube but changes in machining practice did not eliminate the cracks. When initial changes in the deposition conditions failed to eliminate these cracks, it was decided to motor test a typical insert containing a delamination crack at the exit end. The microstructure of both the inlet and exit end of the coated insert selected for the first subscale firing is shown in Figure 7. The crack in the exit end which averaged about 1 mil in width and was about 10 to 15 mils above the substrate is typical of all those observed in the subscale insert deposition work.

The cracking observed in the initial coatings on subscale inserts was always at the exit end of the insert and seemed to be aggravated by the machining operation following deposition. Therefore, a modification of the substrate design was made so that no machining was required at the exit end of the coated section following deposition. A small wrap-around section of the coating at the exit end prevented microscopic examination of the coating there, but a partial crack at the inlet end of the test section was noted. To compare the effect of the stress relief crack at the inlet with the crack at the exit end, this insert was selected for the second subscale motor test.

The next modification in the deposition work for subscale inserts consisted of the use of an extruded graphite substrate. The improved match of thermal expansion coefficients in the axial direction was expected to improve the crack resistance of the coating. A flaw-free coating was prepared on an extruded graphite substrate, but during final machining on the outside diameter of the substrate, a hoop tensile failure occurred in the substrate because of the lower strength of the extruded graphite in this direction. This substrate failure propagated into the coating and caused it to crack also. At this point in the subscale program, the use of extruded graphite substrates was set aside in favor of other process modifications. Only a limited number of subscale inserts were required so that success with other process modifications prevented the return to the study of extruded graphite. Thus, the possibility of preparing coatings on subscale substrates of extruded graphite was not fully exploited.

Success had been achieved in an earlier program (Ref. 1) in preparing crack-free inserts of a size almost identical to the current subscale design. A re-study of these deposits indicated a subtle variation in coating structure especially in the exit end of the nozzle where the current problem with delamination flaws was most prevelant. By following this line of attack and making further changes in the process conditions, a steady



a. Entrance (X150)



b. Exit (X150)

Figure 7. Microstructure of Pyrolytic Graphite Coating on Insert for Subscale Firing Number 1.

advance in our capability to prepare flaw-free coatings was made in the subscale system. The first flaw-free coated insert prepared and fully machined for firing had a coating thickness of approximately 23 mils at the throat. The microstructure of this coating, which was successfully tested in subscale Firing No. 3, is shown in Figure 8. The increased renucleation in this coating is apparent compared to that in Figure 7.

Following the preparation of this relatively thin crack-free coating deposition work continued along the same line and crack-free coatings as thick as 54 mils were successfully deposited on subscale molded graphite substrates. The microstructure of the 54-mil coating, tested in subscale Firing No. 4 is shown in Figure 9. The upper limit of coating thickness which can be made by the preferred deposition technique with a molded graphite substrate of the subscale configuration was not determined since the requirements of the motor test program for test nozzles was fulfilled before this limit was reached. The requirement for fullscale inserts then took precedence over further laboratory study on subscale inserts. The fact that discrete coating losses occurred during motor test of the 54-mil-thick coating, as discussed later in this report (Section 5.B.4.), suggests that the upper limit was at hand. The residual stresses plus the stress factors associated with the nozzle conditions apparently led to progressive coating failure.

Prior to the completion of the subscale deposition work, enough study of the deposition process for fullscale inserts was completed to indicate that significant problems could be anticipated in preparing flaw-free coatings of sufficient thickness for full duration motor firings. Thus, the final work on subscale deposition consisted of an exploratory test of the suitability of a low modulus, fibrous graphite as a substrate material. A flaw-free coating 51 mils thick was deposited on a fibrous substrate. More important for the timely pursuit of the program objectives, this coated insert was successfully tested on subscale motor Firing No. 5, as described below in Section 5.B.5.

C. FULLSCALE INSERTS

Deposition studies with fullscale substrates were started during the fourth month of the program and were continued for the remainder of the program. Thus, during the middle of the contract period subscale and full-scale deposition work proceeded concurrently. This approach was adopted so that maximum use could be made of the information gained in the deposition work. Deposition procedures suitable to only one size insert were thus avoided.

In the preparation of fullscale inserts the substrate system was segmented so that the insert was separate from the inlet and outlet sections. This design reduced the amount of machining required on the coated insert and thus largely eliminated this possible source of damage to the coatings.

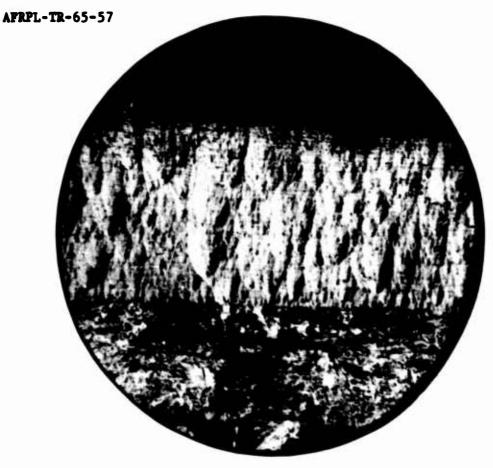


Figure 8. Microsturcture of Pyrolytic Graphite Coating at Entrance of Insert for Subscale Firing Number 3 (X150).

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a. Entrance (X60)



b. Exit (X150)

Figure 9. Microstructure of Pyrolytic Graphite Coating on Insert for Subscale Firing Number 4.

For the larger scale pieces this design also provided a significant cost reduction in preparation of the fixtures.

In the initial deposition work with fullscale inserts, delamination cracks were observed at coating thickness of the same order of magnitude as those which led to cracks in the subscale coatings. At first this was somewhat surprising since, in general, the principal stresses caused by coating anisotropy are roughly a function of the ratio of coating thickness to radio of curvature rather than to the absolute value of the coating thickness. However, stresses produced by mismatch of substrate and coating expansion properties are more nearly dependent on the linear measurements of the composite. Thus, the observed behavior suggests a dominate role of thermal expansion stresses in the delamination phenomenon.

A qualitative observation of note made on the early fullscale inserts was that the nature of the cracking was somewhat different from that seen on the subscale inserts. On the fullscale inserts cracking occurred largely at or very near the substrate-coating interface. On the subscale inserts the cracks were generally completely within the coating, most often two-tenths or more of the coating thickness away from the interface.

Prior to the achievement of flaw-free fullscale coatings of usable thickness, one insert containing a typical microscopic delamination separation at the exit end was selected for motor test. The microstructure of this coating at each end is shown in Figure 10.

The observations regarding delamination cracks in fullscale coatings led to emphasis on two factors in the deposition work. First, to achieve maximum bond strength a thermal pretreatment of the substrate was adopted. This pretreatment was performed following all machining operation just prior to the deposition run. A sooty layer on the surface was found on some inserts following pretreatment and that would likely be detrimental to the coating-substrate bond. The substrate was dry polished following pretreatment to provide a clean deposition surface.

The second factor emphasized to improve the integrity of full-scale coatings was the selection of different grades of graphite as substrates in an effort to improve the compatibility of coating and substrate. The use of an extruded graphite (Speer 580) provided sufficient relief for the incompatibility problem (principally in the axial or with-grain direction) so that a flaw-free coating 47 mils thick was prepared. The microstructure of this coating, which was motor tested in fullscale Firing No. 2, is shown in Figure 11. A similar coating of 64 mils thickness on the Speer 580 contained a delamination crack.

To achieve still thicker coatings without cracks, substrates were machined from a low modulus, fibrous graphite (National Carbon PT-0114).

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a. Entrance (X150)



b. Exit (X150)

Figure 10. Microstructure of Pyrolytic Graphite Coating on Insert for Fullscale Firing Number 1.



a. Fullscale Firing Number 2; Exit (X150)



b. Fullscale Firing Number 3; Exit (X150)

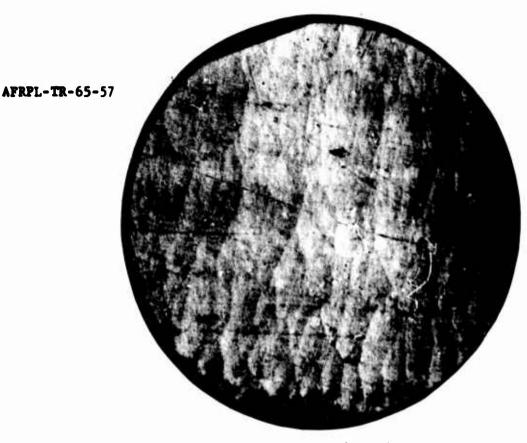
Figure 11. Microstructure of Pyrolytic Graphite Coating on Insert for Fullscale Firings Number 2 and Number 3.

Pyrolytic graphite coatings as thick as 100 mils were successfully deposited on this type of substrate waterial without observable delamination cracks. A coating of 75 mils thickness on the fibrous graphite substrate was selected for test in fullscale Firing No. 3. The microstructure of this coating is shown in Figure 11.

The thickest coating prepared on this program, which was also deposited on the fibrous type substrate, was selected for fullscale Firing No. 4. The microstructure typical of this coating is illustrated in Figure 12. The as-deposited coating, which was 101 mils thick, contained a number of nodules which had grown to various sizes up to the order of a tenth of an inch in diameter. Microscopic examination of the polished edge of the coating revealed that the local flaws in these nodules, which are characteristic of any nodule, were more severe than usual. Because of the surface roughness introduced by these nodules, the surface was hand-polished prior to motor test. To define the effect of this polish ag operation in the area of the local flaws, photomicrographs were made both before and after this smoothing step. Views of typical nodule regions both as deposited and after surface polishing are shown in Figure 13. It is apparent that the polishing did not significantly interact with the pre-existing local flaws except that it allowed some of these flaws to be exposed at the finished surface. The local flaws lie along the locally distorted direction of the pyrolytic graphite layer planes. Polishing the surface in the region of a nodule cuts across the layer planes locally and consequently exposes the edge of the planes in these areas. The role of local flaws in increasing the erosion rate of a pyrolytic graphite coating was not determined quantitatively, but qualitatively it can be surmised that some loss in erosion resistance is inevitable. This assumption was borne out in the results of the fourth fullscale firing described later. Optimization of the coating structure and reduction of the module population were beyond the scope of this feasibility program but the incentive for such an effort was indicated.

D. CORRELATION OF RESIDUAL STRESSES AND OBSERVED CRACKS

In the preceding major section of this report, the residual stresses calculated for coated composites were presented. In this section, the experimental evidence of delamination cracking was outlined. In Table VI, the data pertinent to the establishment of failure stress criteria are summarized. Two calculated stress values, the radial tension in the coating and the axial tension in the substrate, are listed for the thickest crack-free coatings and the thinnest coatings which contained cracks for each substrate material used. As outlined in the introduction to the stress analysis discussion, the two types of tensile stress listed are those which are consistent with the nature of the cracks observed. Examination of Table VI indicates failure criteria which are consistent with all the



a. Entrance (X150)



b. Exit (X150)

Figure 12. Microstructure of Pyrolytic Graphite Coating on Insert for Fullscale Firing Number 4.



a. As Deposited (X60)



b. After Surface Polishing (X60)

Figure 13. Local Flaws Associated with Nodules in Coating on Insert for Fullscale Firing Number 4.

Table VI.

Correlation of Calculated Stresses

and Observed Delamination Cracks

Substrate Type A. Subscale, 1.72	Coating Thickness (mil) inch Diameter	<u>Cracks</u>	Calculated Radial in Coating (psi)	Stresses* Axial in Substrate (psi)
ATJ	54	No**	100	<u>950</u>
Fibrous	51	No	280	260
B. Fullscale, 2.	30 inch Diameter			
ATJ	22	No	Compressive	520
ATJ	42	Yes	Compressive	920
580	47	No	220	520
580	64	Yes	340	510
Fibrous	100	No	230	400

^{*}Interpolated and extrapolated from data in Table V.

No cracks observed, but coating failed during motor test.

experimental evidence gathered. The stress values underlined in the table are called to the reader's attention as those which appear critical. A radial tensile stress level in the coating between 280 and 340 psi separates the crack-free and cracked condition. An axial tensile stress in the substrate between 520 and 920 psi appears critical. In the case of the 54-mil coating on the subscale ATJ substrate, no cracks were observed although the axial tension in the substrate was calculated to be 950 psi. However, this coating failed and was removed during motor test which suggests a residual stress condition close to the failure level.

Although the absolute values of the failure stresses indicated are subject to the approximations involved in the calculations, the discovery of a self-consistent body of calculated and experimental evidence on delamination cracking is significant. Optimization of fabrication technology can be pursued by reduction of the residual stresses or by increasing the tolerance of the pyrolytic graphite for these stresses. The advances achieved in the deposition work on this program came from work along both of these paths, but further advances should be possible beyond the scope of this program.

5.0 MOTOR FIRING TESTS

The focal point of the entire program to determine the feasibility of pyrolytic graphite coated nozzle inserts was the motor firing evaluation. To prove the feasibility of a nozzle material, critical motor firing tests must be completed successfully. The goal of this program was to prove the suitability of a pyrolytic graphite coated nozzle for service with a 6550°F propellant and a motor pressure of 700 psi in a reasonably size system and for a realistic firing duration. To achieve this goal, in addition to the analytical and deposition work described elsewhere, a series of subscale and fullscale motor firings was carried out. The subscale motor firings using a nozzle of 1.1-inch throat diameter for a motor of approximately 1100 pounds thrust were carried out to define the suitability of the basic design and material for critical nozzle service. Following the subscale effort, a series of fullscale firings using a nozzle of 2.3-inch throat diameter with a motor of approximately 4600 pounds thrust was carried out to demonstrate usable service in sufficiently large nozzles to be indicative of the merit of pyvolytic graphite coating. In the following sections, the motor firing procedures and the results of the subscale and fullscale firings are discuszed separately.

A. TEST PROCEDURE

The motors used for both subscale and fullscale nozzle tests were of an insulated heavy-weight design and were filled with gel propellant. The use of gel propellant and a heavy-weight motor design leads to a minimum cost program of nozzle materials evaluation. The propellant used in all firings in this program was a highly aluminized solid propellant having a theoretical flame temperature of 6550°F and moderately oxidizing combustion products. This propellant is designated at Atlantic Research as APG-112. The characteristics of this propellant are summarized in Table VII.

Each test nozzle was fabricated and assembled in our laboratory and shops and then transported to the rocket test bay. Figure 14 is a photograph of the fullscale test motor in operation. The level of the motor pressure and changes in the motor pressure along with pre- and post-firing measurements and examinations define the erosion performance of a nozzle material. Thermocouple instrumentation was placed on the first nozzle tested to confirm the design selection but temperature instrumentation was not used on later tests.

B. SUBSCALE MOTOR FIRINGS

A total of five subscale evaluation tests were successfully completed. The design philosophy for both the subscale and fullscale nozzles utilized the segmented concept in which the pyrolytic graphite coated insert was only a segment large enough to provide dimensional stability at the throat. A number of other segments were used to form the total nozzle contour and support, back-up, and insulate the exposed segments. The basic design used in all of the subscale nozzles is shown in the

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Table VII. Characteristics of APG-112 Propellant

	CHAMBER	EXHAUST
Specific Impulse (lb-sec/lb)	262.4	
Characteristic exhaust velocity (ft/sec)	5042	
Temperature (°K)	3894	2719
Specific Heat (cal/100;-°K)	44.26	42.96
Moles of gas (g moles/100g)	2.9833	2.7849
Pressure (psia)	1000	14.699
Combustion Products g moles/100g		
C	0.0000	0.0000
н	0.2678	0.1057
0	0.0074	0.0003
N	0.001	0.0000
C1	0.0648	0.0333
A1	0.0121	0.0003
CO	0.5403	0.5465
∞_2	0.0191	0.0128
CN	0.0000	0.0000
HCN	0.0000	0.0000
н ₂	0.9790	1.1281
H ₂ 0	0.2909	0.1758
HC1	0.3494	0.4904
02	0.0009	0.0000
OH	0.0492	0.0048
N ₂	0.2659	0.2682
МО	0.0042	0.0002
NH	0.0003	0.0000
NH ₂	0.0001	0.0000
NH ₃	0.0000	0.0000
Cl ₂	0.0001	0.0000

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Table VII. (Continued)

Combustion Products		
Alh	0.0017	0.0000
A10	0.0045	0.0000
A1 ₂ 0	0.0023	0.0000
A1202	0.0003	0.0000
A1C1	0.1112	0.0168
AlCl ₂	0.0051	0.0010
AlCl ₃	0.0006	0.0002
A10C1	0.0059	0.0004
C (solid)	0.0000	0.0000
Al ₂ 0 ₃ (liquid)	0.4335	0.4973
Al ₂ 0 ₃ (solid)	0.0000	0.0000
AlN (solid)	0.0000	0.0000

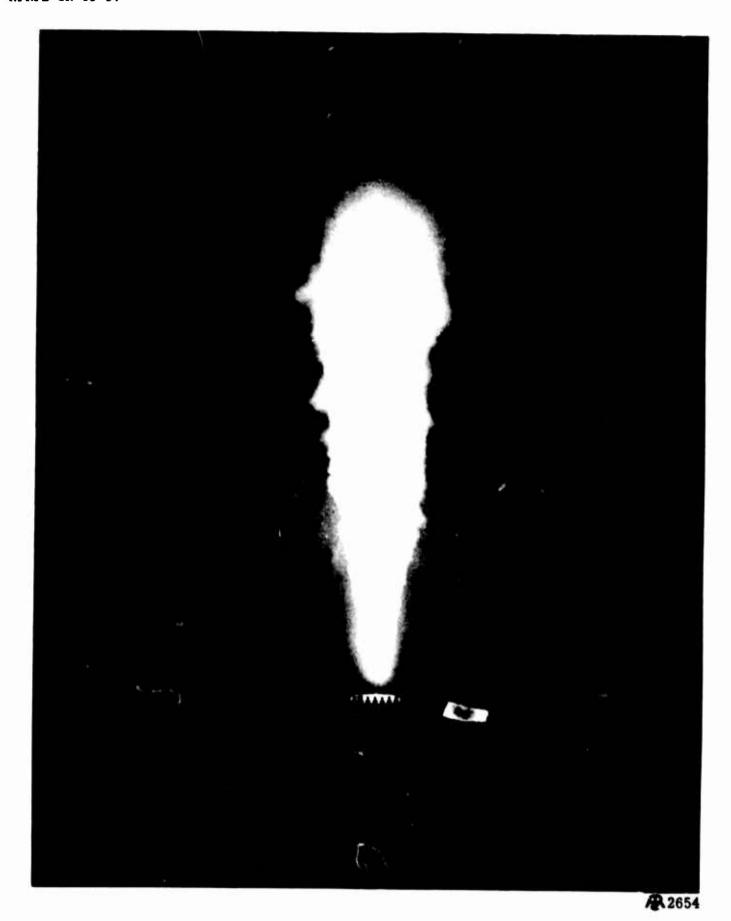


Figure 14. Fullscale Test Motor During Firing.

assembly drawing, Figure 15. The actual segments used in one of the subscale nozzles are pictured prior to assembly in Figure 16.

For the subscale nozzles the radius of curvature of the insert substrate was chosen to be approximately three times the throat radius. The length of the coated insert was selected so that the joint line at the inlet end was at an area ratio of 2 relative to the throat, and the exit joint line occurred at the 15° tangent point to match the expansion cone half angle. Previous work had indicated the desirability of protecting the inlet edge of the coated insert from excessive exposure to the flow of combustion gases. For convenience in this program, a single plate of pyrolytic graphite in the edge-oriented position was placed immediately upstream of the pyrolytic graphite coated throat insert. In a flight-weight nozzle design, the optimum selection for the entrance section might well be a second pyrolytic graphite coated segment. For the test nozzles in this program the pyrolytic graphite plate and the insulation thicknesses were selected to provide nozzle integrity in accordance with the results of the thermal analysis discussed in another section.

To provide for the thermal expansion of the exposed segments in each test nozzle, calculations were made of the expansion to be expected from each segment so that expansion joints could be installed at selected locations. The total expansion of each of the exposed segments shown in the assembly drawing, Figure 15 was calculated as follows:

- (1) Entrance section 1 19 mils.
- (2) Entrance section 2 25 mils.
- (3) Throat section 16 mils.
- (4) First exit section 18 mils.
- (5) Exit cone 39 mile

To accommodate these expansions the following design decisions were made:

- (1) The expansion of the first inlet section would be taken by the shrinkage and crushing of the reinforced plastic at the nozzle entrance.
- (2) An expansion joint 30 mils thick was placed at the entrance end of the second entrance section to accommodate 50 per cent of the total expansion of the second, third and fourth segments of the nozzle. A silicone rubber expansion gasket was used at this upstream location in all tests.

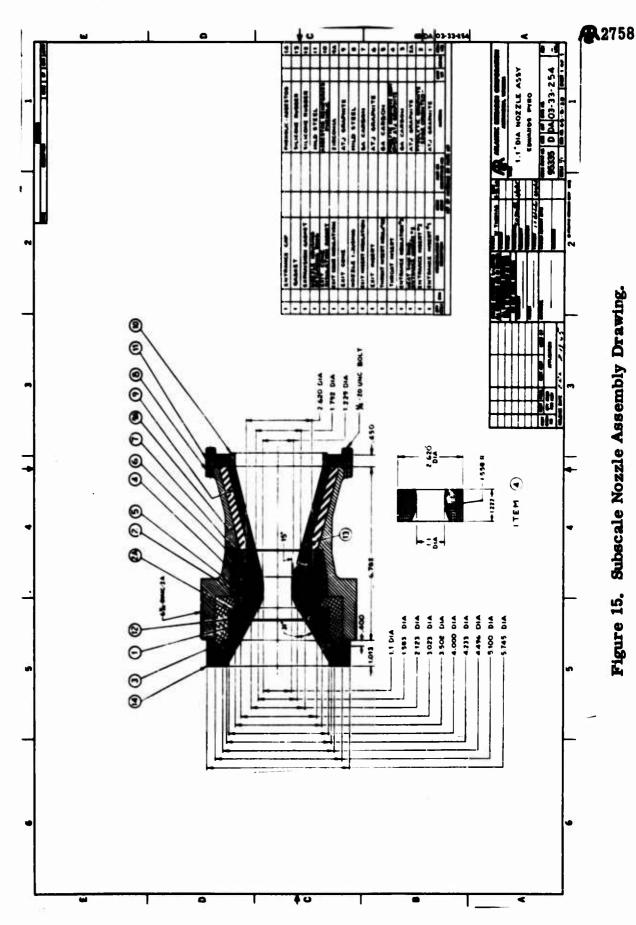
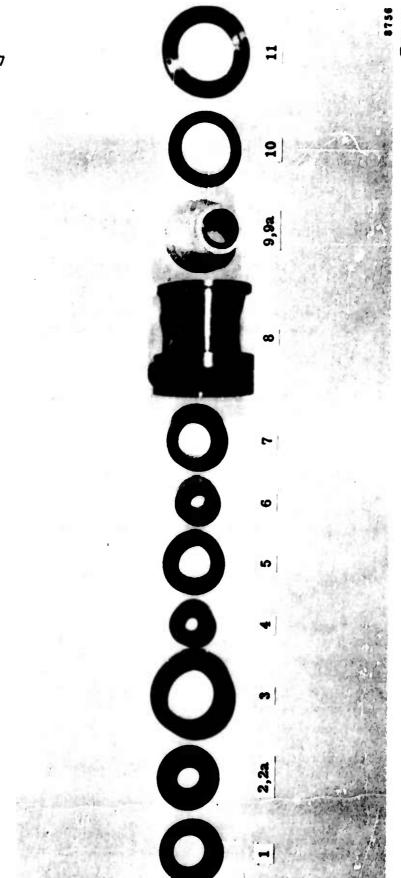


Figure 15. Subscale Nozzle Assembly Drawing.

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(3) A second expansion joint between the fourth and fifth segment might be required to accommodate the expansion in this region. The second expansion joint utilized in the first test was found unnecessary and eliminated in subsequent tests. (U)

A summary of all of the evaluation data obtained in the motor test firings is contained in Table VIII. In the following sections, each of the subscale firings is described and discussed separately, and then a comparison and discussion of the implications of these firings is presented. (U)

1. Subscale Firing No. 1

The first subscale firing was carried out prior to the point in the deposition study when delamination flaws were eliminated. The insert selected for this test had a coating thickness of 58 mils at the throat and contained a microscopic delamination at the exit end of the insert. Nozzle erosion during this test was smooth as can be seen from the motor pressure trace in Figure 17, but the rate of erosion was greater than that predicted for a good coating. The erosion was sufficient to remove the entire coating during the 56 second duration at an average motor pressure of 564 psi. Figure 18 shows that although insert erosion was higher than desirable, the general condition of the remainder of the nozzle was satisfactory. (C)

The over-all average radial erosion rate calculated from preand post-firing throat dimensions was 1.91 mils per second. Post-firing examination of the nozzle and ballistic calculations based on the motor pressure trace suggest the following series of events likely took place in this test. (C)

A significant initial loss of coating occurred within the first 5 seconds or so, probably at the exit end of the insert. For a major portion of the firing, erosion proceeded by minor spallation and edge chipping at the exit end of the coating to yield an average erosion rate of about 1.2 mils per second. During this period the location of the throat plane apparently moved upstream. Finally more rapid erosion occurred near the end of the firing as a greater amount of the substrate graphite was exposed. In Figure 19 the estimated throat radii during this firing are shown as determined by a ballistic calculation from point to point along the motor pressure curve. It was indicated by this firing that the pyrolytic graphite coating was not likely to be tolerant of delamination flaws under service conditions so severe as used in this program. (C)

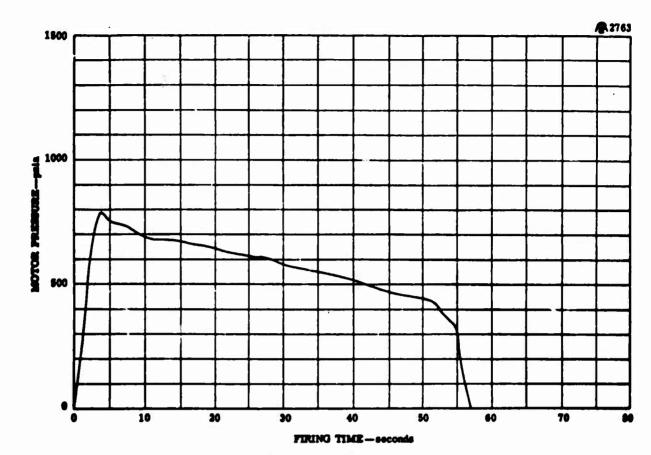
2. Subscale Firing No. 2

The insert tested in the second subscale firing had a wraparound coating contour at the exit end, but it still contained a partial (C)

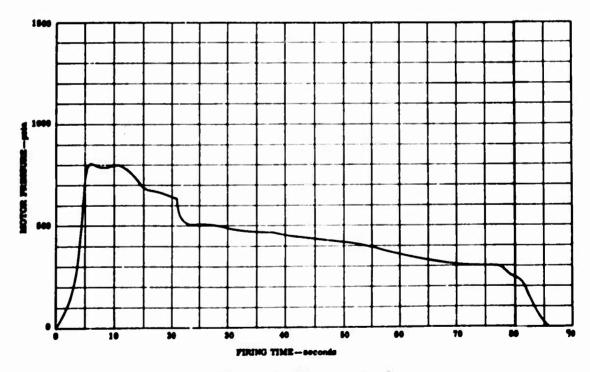
Table VIII. Motor Test Data Summery

		Insert	Insert Description	Fir	Firing Conditions	800		Performance Data	Į.
Program Test No.	A.R.C. Firing Designation	Substrate	Coating Thickness (mil at throat)	Duration (sec)	Max. Mtr. Pressure (psi)	Avg. Mtr. Pressure (psi)	Throat dia. before feat (inch)	Throat dia. after Test (inch)	Avg. Radial Erosion Race (mil/sec)
SS-1	11	TŞ.	58	55.9	795	. 35	1.105	1.287/1.351	1.91
SS-2	EPb-2	Ą	99	83.0	807	452	1.090/1.097	1.485/1.510	2.43
\$8-3	E-9-3	AE.	23	¥.8	717	618	1.136	1.173/1.176	0.55
7 55	2-4	A <u>t</u>	*	6.8	812	290	1.111	1.330/1.365	1.82
\$-\$\$	9-44	Fibrous	51	37.3	585	530	1.190	1.227/1.249	3.0
75-1	7-923	A.	AEJ 42	36.6	723	623	2.316	2.429/2.493	2.01
15-2	E-1	280	1.7	31.5	702	1	2.306	2.342/2.358	0.73
15-3	8-9Z	Fibrous	7.5	42.8	111	269	2.259	2.328/2.342	0.89
7 22	6-94	Fibrous	66	63.5	867	108	2.242	2.393	1.19

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Subscale Firing No. 1



Subscale Firing No. 2

Figure 17. Motor Pressure Traces for Subscale Firings No. 1 and No. 2.

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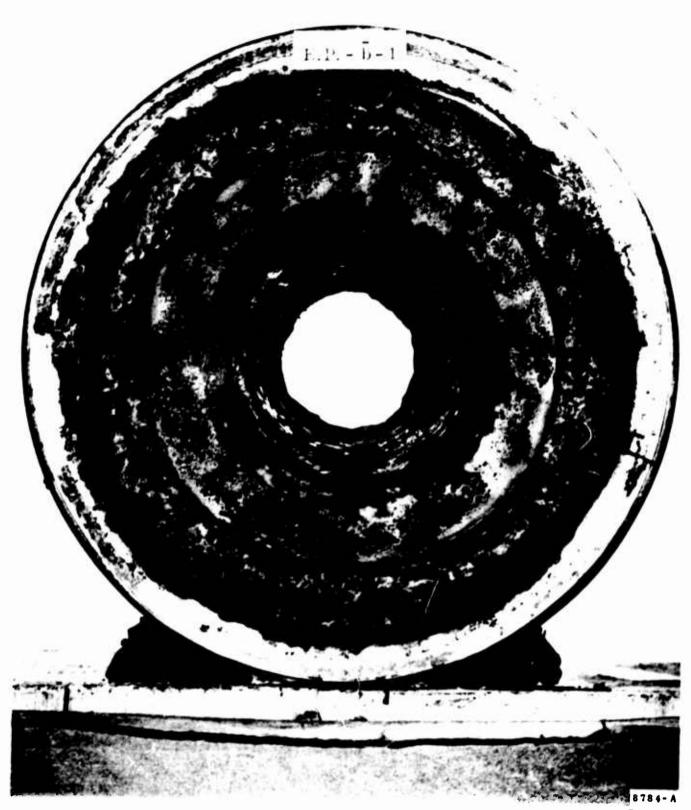


Figure 18.. Axial View from Entrance End of Nozzle After Subscale Firing No. 1.

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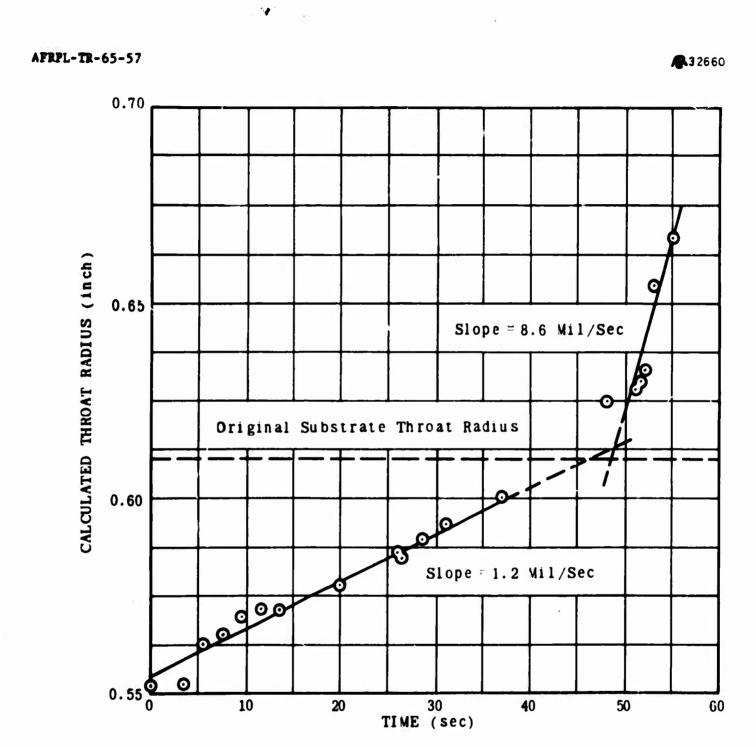


Figure 19. Calculated Throat Radii During Subscale Firing No. 1.

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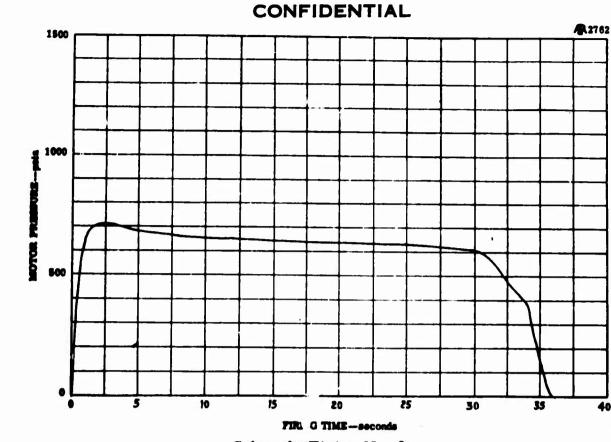
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delamination flaw at the entrance end. The performance of this insert was poor and reinforced the belief that coatings are not tolerant of delamination flaws under severe service conditions. The pressure-time trace for this firing (Figure 17) shows clear evidence of the loss of sections of the coating during test. The total firing duration was 83 seconds and the coating was completely removed during test. The average radial erosion rate calculated from pre- and post-test measurements was 2.43 mils per second which clearly indicates the combined erosion of the coating and later the substrate. The relatively smooth pressure regression that the exception of the sudden pressure losses associated with coating spallation suggests that the coating eroded progressively from the downstream edge towards the entrance and provided some protection for the substrate during much of the firing period. (C)

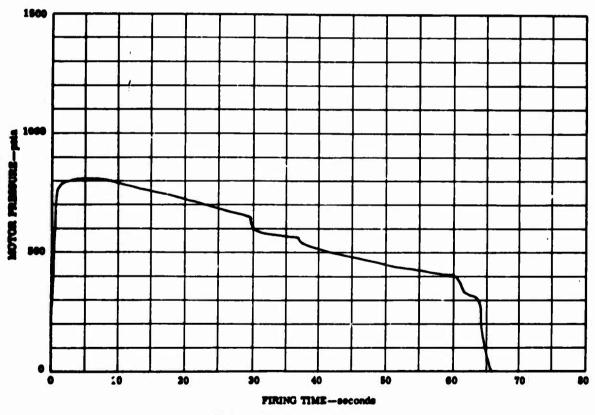
3. Subscale Firing No. 3

The third subscale firing was the first test of a pyrolytic graphite coated insert which showed no visible cracks upon microscopic examination of both ends of the insert. The average erosion rate of 0.55 mil per second at an average motor pressure of 618 psi demonstrated the basic serviceability of a pyrolytic graphite coating when the coating integrity is maintained. The coating thickness on the insert tested was only 23 mils at the throat so that the firing duration was reduced to 34.8 seconds. The motor pressure trace shown in Figure 20 reflects the excellent performance of this nozzle. (C)

A most interesting and unique result of the firing test on the coating was observed in post-firing microscopic examination. The integrity of the coating was in no way decreased by the firing, but clear evidence of some plastic deformation of the pyrolytic graphite coating was found. This evidence for deformation is shown in two views of the post-firing microstructure in Figure 21. Markings in the coating resembling slip bands were found at numerous points in the coating. The lower view in Figure 21 includes a void area generated in the substrate beneath the coating which appears to have altered the point at which the two deformation bands intersect probably due to the reduced local stiffness of the substrate. The most significant conclusion to be drawn from these photomicrographs is that a pyrolytic graphite coating appears to be an excellent candidate for restart nozzle service. Further evidence is shown later to reinforce the conclusion that motor firing conditions do not degrade the remaining coating quality. The evidence of plastic deformation without cracking suggests that some relief may occur which could improve the stress distribution during recycle duty. (U)



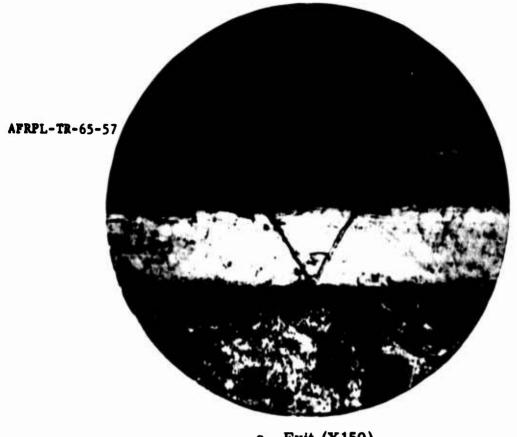
Subscale Firing No. 3



Subscale Firing No. 4

Figure 20. Motor Pressure Traces for Subscale Firings No. 3 and No. 4.

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a. Exit (X150)



b. Exit (X150). Note Void in Substrate.

Figure 21. Microstructure of Pyrolytic Graphite Showing Evidence of Deformation After Subscale Firing Number 3.

4. Subscale Firing No. 4

This firing was carried out to demonstrate the erosion resistance of a thicker coating in substantiation of subscale firing No. 3. The insert tested consisted of a 54-mil thick coating of pyrolytic graphite on a molded graphite substrate. The performance of this insert was disappointing. Discrete losses of coating occurred first at about 29 seconds and again at 37 seconds. The motor pressure trace for this firing is shown in Figure 20. The duration of this firing was 64.9 seconds and the average motor pressure was 590 psi, reduced to this level from an initial maximum motor pressure of 812 psi by an erosion rate averaging 1.82 mils per second. (C)

Although ballistic calculations indicate that the erosion rate of the coating prior to the first discrete loss of coating at about 29 seconds was only 0.7 to 0.8 mil per second, the failure of the coating was serious, but it was the only instance of loss of a crack-free coating in the entire program. It must be concluded that in this insert the cumulative effect of residual stresses from deposition and the thermal stresses generated during the firing caused progressive coating failure. Although this behavior may have resulted from an undetected flaw in this particular insert, time did not permit further study of the optimization of coatings on subscale molded graphite inserts. Instead, a more immediate means of reducing the stress effects was sought through the choice of another substrate material. The extent to which stress control can be achieved through the use of a low modulus substrate is discussed in the section on stress analysis, and the success of this approach was demonstrated in the next subscale firing. (C)

Subscale Firing No. 5

The coated insert tested in this firing consisted of a 51-mil thick py ytic graphite coating on a low modulus fibrous graphite substrate. During the 37 second duration at an average motor pressure of 530 psi, the coating erosion rate averaged 0.64 mil per second. The pressure trace is reproduced in Figure 22. This firing was significant for two reasons. It again demonstrated that a coating without observable flaws could give good performance under severe nozzle conditions. Second, it illustrated that the low modulus fibrous graphite could be successfully utilized as a substrate and survive not only residual stresses involved in deposition, but also the rigors of motor firing. Demonstration of this second fact was particularly important prior to the use of such a fibrous graphite substrate for fullscale firing tests. A photograph of the nozzle assembly after test shown in Figure 23 confirms the good condition of the nozzle. (C)

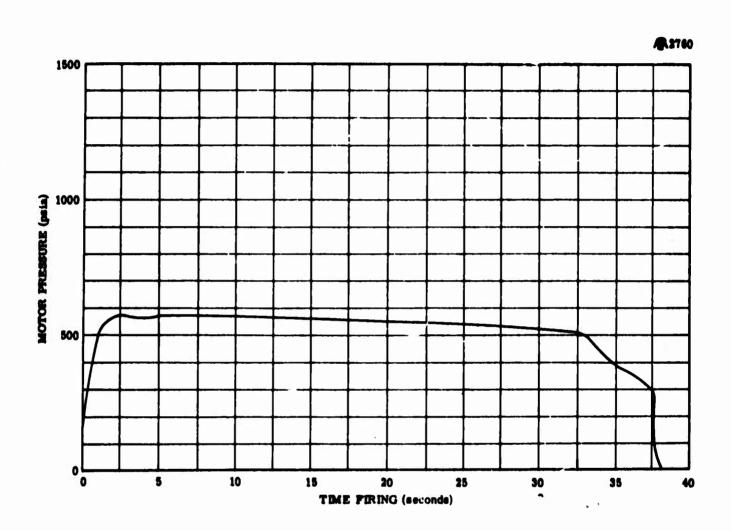


Figure 22. Motor Pressure Trace for Subscale Firing No. 5.

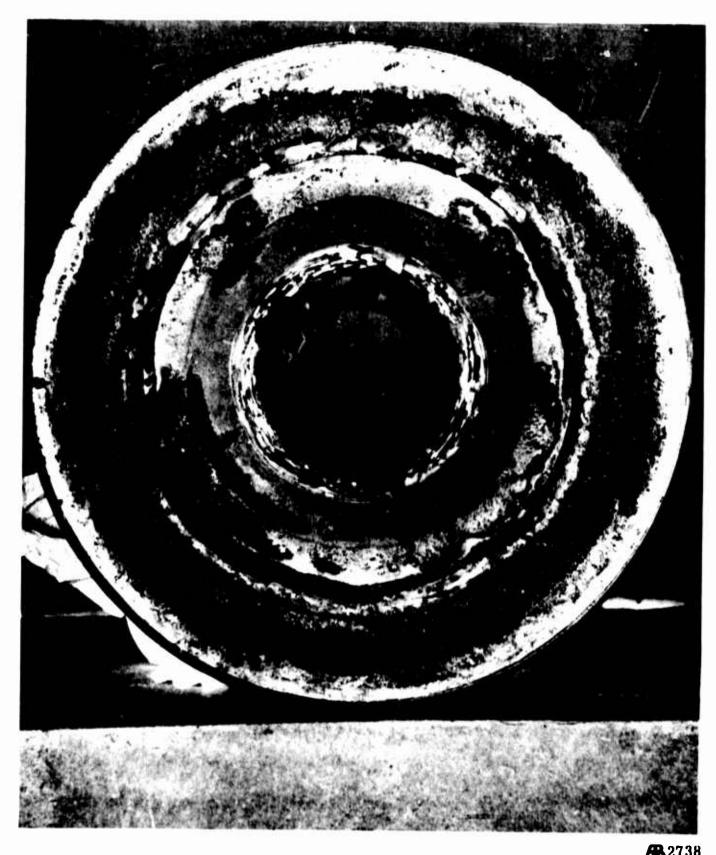


Figure 23. Axial View from Entrance End of Nozzle After Subscale Firing Number 5.

6. Implication of Subscale Test Results

The subscale firings in this program were intended to define problem areas in pyrolytic graphite coatings and to insure the successful demonstration of the feasibility of such coatings for fullscale nozzles. From the results of the five subscale firings, the following conclusions were drawn. The tolerance of pyrolytic graphite coatings for delamination flaws is low. However, it was also indicated that coatings free from visible flaws in microscopic examination could yield excellent nozzle performance for coatings on both molded substrates and low modulus fibrous substrates. The losses from the 54-mil thick coating tested in the fourth subscale firing, reinforces the belief that coating integrity is related to the deposition residual stresses. Although no flaws were visible in this insert, it is apparent that the coating was near the upper limit for flaw-free deposition and it is likely that the built-in stresses led to failure when firing stresses were superimposed on the insert. (C)

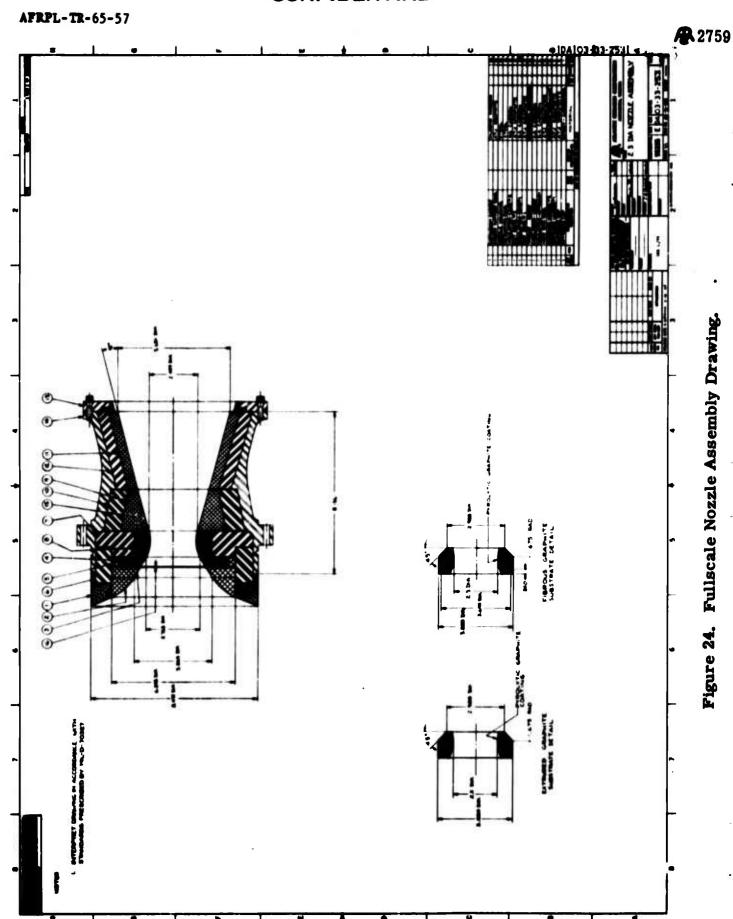
C. FULLSCALE MOTOR FIRINGS

Four fullscale firing tests using a 2.3-inch nozzle diameter in a 4600 pounds thrust motor were carried out in this program. The fullscale nozzle design which is shown in Figure 24 was similar to that used for the subscale unit. The goal of these firings was to demonstrate the performance capability of pyrolytic graphite coatings in nozzles of a usable and practical size. Each of the fullscale firings is discussed separately in the following sections and the implications of these results are indicated. (U)

1. Fullscale Firing No. 1

The fullscale firing program overlapped the subscale program in the same way that the deposition work for the two size ranges overlapped. This overlap was used to assure that results derived for subscale nozzles would be applicable to fullscale systems. The inserts selected for the first fullscale firing consisted of a 42-mil thick coating on molded graphite and contained a visible microscopic separation of the coating from the substrate at the exit end. The nature of the flaw in this insert was quite different from those found in early subscale units. A test of the effect of such a flaw on fullscale nozzle performance was deemed desirable. This firing was made prior to the point in the deposition study when flaw-free fullscale coatings were prepared. (U)

The performance of this fullscale insert with a known delamination flaw was quite similar to the results obtained previously in the subscale test with an insert with a flaw at the exit end. The over-all erosion rate, based on before-and after-test diameter measurements was 2.01 mils per second. The motor pressure trace shown in Figure 25 indicated a steady but excessive erosion pattern. The firing duration (C)

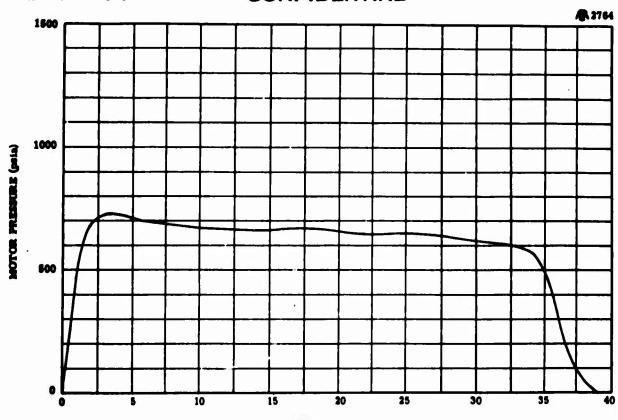


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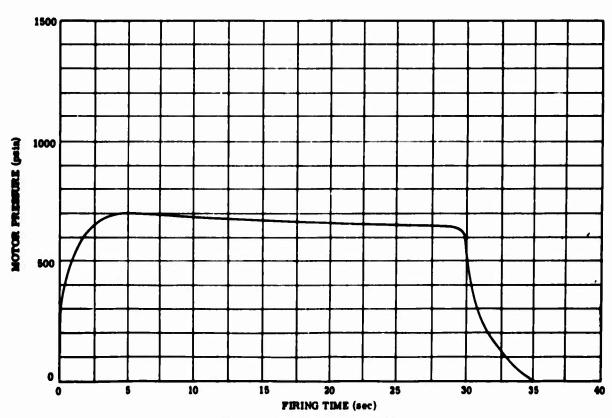


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FIRING TIME (sec)

Fullscale Firing No. 1



Fullscale Firing No. 2

Figure 25. Motor Pressure Traces for Fullscale Firings No. 1 and No. 2.

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was 36.6 seconds at an average motor pressure of 623 psi. A summary of motor test data for the fullscale firings is included in Table VIII. (C)

The consistent failure of coated inserts with known flaws to provide suitable performance suggested that flaw-free inserts were a must to achieve optimum performance. (U)

2. Fullscale Firing No. 2

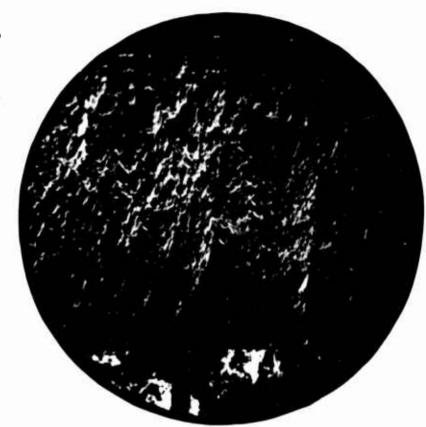
Prior to fullscale Firing No. 2, successful deposition of a flaw-free coating on a Speer Carbon extruded graphite substrate (Grade 580) was achieved. The second fullscale test utilized such an insert with a 47-mil thick coating. This firing was completely successful. The insert showed an average erosion rate of 0.73 mil per second and an average motor pressure 644 psi for a firing duration of 31.5 seconds. The motor pressure trace for this test is included in Figure 25. After test the coating was intact and in good condition with approximately one-half of the original coating thickness remaining. (C)

Further evidence of the excellent condition of the coating remaining on the insert after this test was found by examining the microstructure of the coating. This microstructure, shown in Figure 26, is in every way similar to that of the insert prior to test and shows no indication of damage or cracking. (U)

3. Fullscale Firing No. 3

The deposition work indicated that a low modulus, fibrous graphite was an interesting alternative to the Grade 580 extruded graphite substrate. The final subscale firing had indicated that a pyrolytic graphite coating on a fibrous graphite substrate was suitable for motor service. To confirm this performance at the fullscale, a coating 75 mils thick deposited on a fibrous graphite substrate was tested in the third fullscale firing. The nozzle gave good performance. An average erosion rate of 0.88 mil per second was measured for an average motor pressure of 697 psi and a firing duration of 42.8 seconds. The motor pressure trace for this firing is shown in Figure 27. (C)

After test, the coating was intact and in generally good condition. Approximately one-half of the original thickness of the coating remained. Two views of the entrance end of the nozzle after test are shown in Figures 28 and 29. These figures indicate the good condition of the entire assembly. The microstructure of the coating after firing shown in Figure 30 again demonstrates that no discernable change occurs in the coating because of the motor exposure. (U)

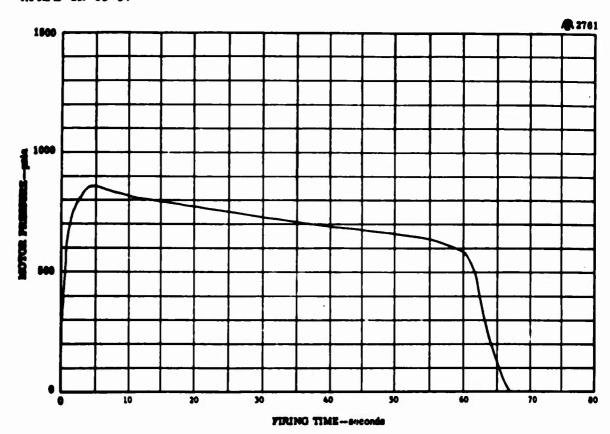


a. Entrance (X150)

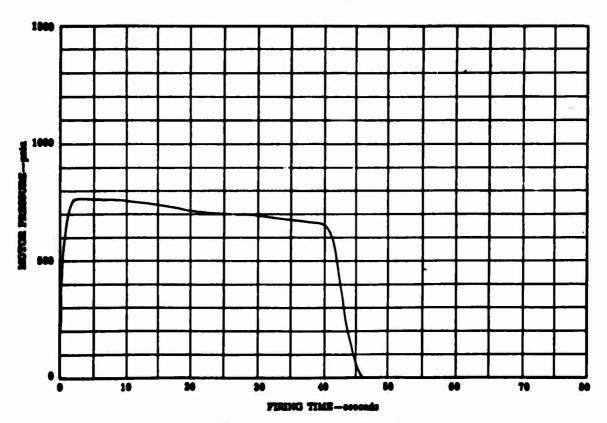


b. Exit (X150)

Figure 26. Microstructure of Pyrolytic Graphite Coating After Fullscale Firing Number 2.



Fullscale Firing No. 3



Fullscale Firing No. 4

Figure 27. Motor Pressure Traces for Fullscale Firings No. 3 and No. 4.

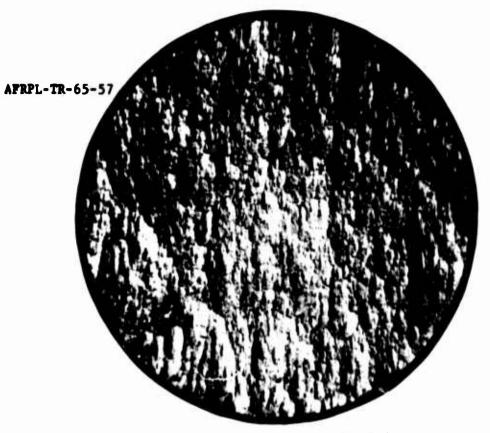
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Figure 28. Axial View from Entrance End of Nozzle After Fullscale Firing No. 3.



Figure 29. Oblique View from Entrance End of Nozzle After Fullscale Firing No. 3.



a. Entrance (X150)



b. Exit (X150)

Figure 30. Microstructure of Pyrolytic Graphite Coating After Fullscale Firing Number 3.

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4. Fullscale Firing No. 4

In selecting an insert for this final fullscale motor firing, the alternative of using an extruded graphite or a fibrous graphite substrate was available. As discussed earlier in the deposition study, coatings of sufficient thickness were not successfully prepared on the extruded substrate. Time did not permit the further pursuit of the deposition study, so an insert consisting of a 100-mil coating of pyrolytic graphite on a fibrous graphite substrate was selected for test. The goal of this firing was to demonstrate a full duration capability of more than 60 seconds for a pyrolytic graphite coated nozzle under the severe motor conditions selected for this program. This goal was achieved in fullscale Firing No. 4 which had a firing duration of 63.5 seconds and an average motor pressure of 708 psi. The motor pressure trace for this test is shown in Figure 27. (U)

Although this firing represented a successful full duration test, the success was qualified by two factors. First, although the motor pressure trace was smooth, a local gouge occurred in the nozzle coincident with motor tailoff. This gouge, which because of the soft and non-erosion resistant nature of the fibrous substrate is sizeable, is seen in the two views of the post fired nozzle in Figures 31 and 32. Second, the average erosion rate calculated from post-firing measurements in areas away from the local gouge indicated a higher than normal erosion rate of 1.19 mils per second. Both of these qualifications on the success of this firing are believed to have resulted from the non-uniform quality of the coating. As indicated in the deposition study section, the coating tested in this firing had more than normal roughness associated with nodules in the coating. To avoid effects associated with turbulence due to surface roughness, the surface of the coating was polished prior to test. However, this smoothing operation could not eliminate the effect of local flaws in nodules on the coating erosion. It is likely that enhanced erosion occurred in the regions where local flaws in the nodules became exposed to the surface. Although the local flaws associated with nodules in the coating probably produced microspallation of the coating locally, the microstructure shown in Figure 33 indicates that this type of flaw does not lead to catastrophic changes in the coating integrity. In Figure 33 the presence of submerged local flaws in the coating remaining after tests indicates this conclusion to be valid. (C)

5. Implications of Fullscale Test Results

The complete loss of the coating of the first fullscale firing again indicates that coatings with known delamination cracks are not suited for severe nozzle applications. The successful completion of the remaining fullscale tests with nozzles that did not contain delamination cracks demonstrated the feasibility of pyrolytic graphite coated nozzles for fullscale applications. Optimization of coatings on fullscale inserts was not within the scope of this program, but the successful performance of three fullscale coated inserts does point to the inherent advantage of this light-weight nozzle system. (U)

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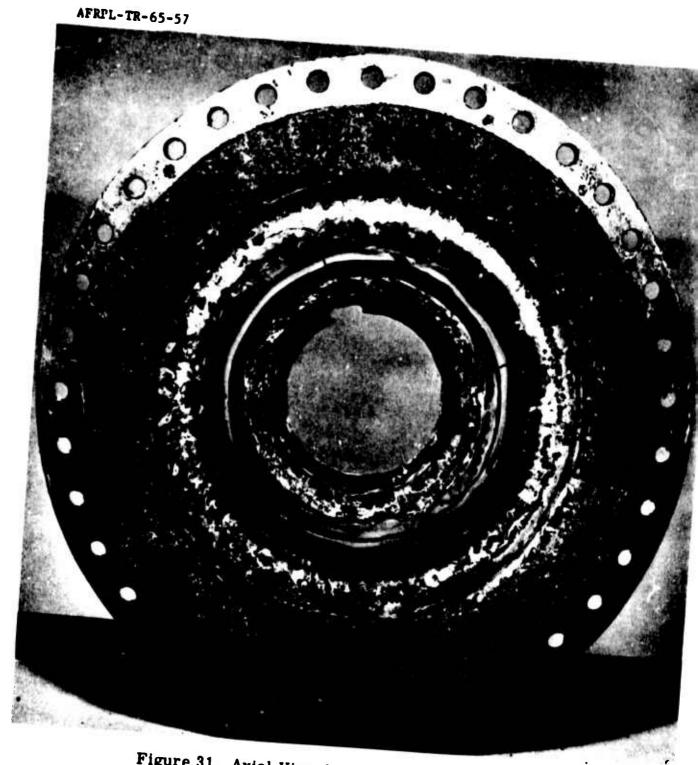
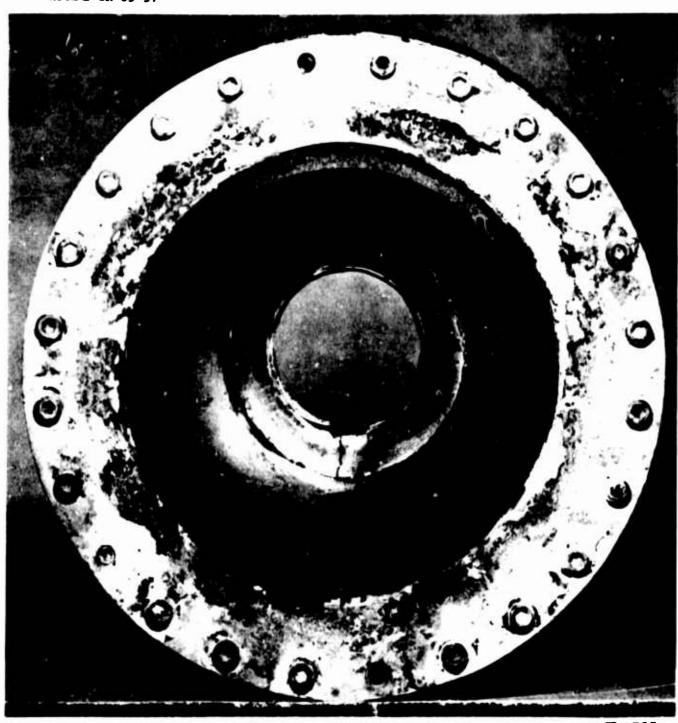
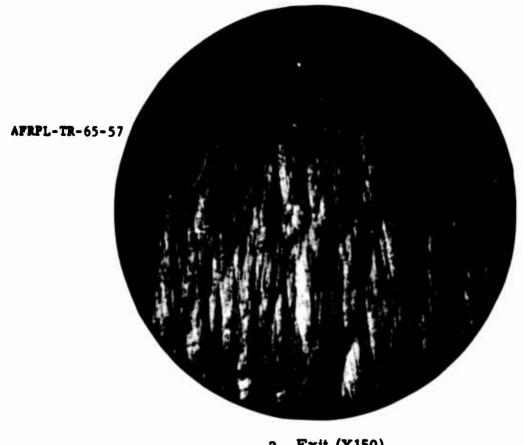


Figure 31. Axial View from Entrance End of Nozzle After Fullscale Firing No. 4.



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Figure 32. Axial View from Exit End of Nozzle After Fullscale Firing No. 4.



a. Exit (X150)



b. Local Flaws in Nodule (X150)

Figure 33. Microstructure of Pyrolytic Graphite Coating After Fullscale Firing Number 4.

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APPENDIX A

Calculated Temperatures in Various Nozzle Designs
Utilizing Pyrolytic Graphite Coatings

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Case A-1
Throat Location: 1.100" diameter

Location Radius Node No.	Surface 0.550 1	Rear Graphite 1.180 11	Midpoint Carbon 1.580 14	Steel Average 2.200 17,18
Time (sec)		Temperature	1	
1	4470	140	70	70
2	4891	315	71	70
5	5330	845	92	70
10	5608	1513	206	70
20	5848	2365	568	79
30	5964	2899	935	106
40	6035	3270	1250	154
5 0	6083	3543	1510	216
60	6117	3751	1725	288

Case A-2
Throat Location: 1.100" diameter

Location Radius Node No.	Surface 0.550 1	Rear Graphite 1.440 13	Midpoint Carbon 1.730 15	Steel Average 2.200 17,18
Time (sec)		Temerature	-7	
1	4430	84	70	70
2	4850	164	75	70
5	5283	533	150	70
10	5550	1114	398	78
20	5798	2004	959	143
30	5932	2654	1443	263
40	6021	3156	1846	413
50	6085	3557	2185	565

Case A-3
Throat Location: 1.100** diameter

Location	Surface	Near Rear PG	Rear Graphite	Midpoint Carbon	Steel Average
Redius	0.550	0.574	1.180	1.580	2.200
Mode No.	1	5	14	17	20,21
Time (sec)			Temeratur	ie. *?	
1	5925	804	85	70	70
2	5959	1119	152	70	70
5	6006	1643	405	78	70
10	6054	2184	780	133	70
20	6113	2866	1350	332	74
30	6151	3326	1772	560	89
40	6178	3620	2041	776	116
50	6199	3858	2365	970	154
60	6215	4046	2582	1142	201

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Case A-4
Throat Location: 1.100" diameter

Location	Surface	Near Rear PG	Rear Graphite	Midpoint Carbon	Steel Average
Redius	0.550	0.574	1.440	1.720	2.200
Node No.	1	5	16	18	20,21
Time (sec)			Temperatur	· · · · · · · · · · · · · · · · · · ·	
1	5925	801	72	70	70
2	5958	1117	94	71	70
5	6004	1611	232	89	70
10	6045	2076	484	174	72
20	6096	2662	910	401	91
30	6129	3057	1247	615	132
40.4	6156	3360	1481	810	192
50.4	6175	3585	1756	974	259
59.4	6190	3749	1929	1107	325

Case A-5
Throat Location: 1.100" diameter

Location	Suiface	Rear PG	Rear Graphite	Rear Carbon	Wear Outside (in Steel)
Radius	0.550	0.610	1.250	2.000	2.220
Node No.	1	6	14	20	21
Time (sec)			Temperature		
.05	4720	70	70	70	70
.1	5130	70	70	70	70
. 2	5450	73	70	70	70
. 5	5782	100	70	70	70
1.0	5955	175	71	70	70
2.2	6081	348	95	70	70
5.2	6134	624	228	70	70
10.2	6152	939	456	70	70
20.2	6176	1406	826	72	71
30.2	6193	1758	1121	79	75
40.2	6207	2038	1364	92	84
50.2	6218	2268	1570	112	100
59.2	6227	2445	1732	135	119

Case A-6
Throat Location: 1.100" dismeter

Location	Surface	Rear PG	Rear Graphite	Rear Carbon	Near Outside (in steel)
Radius	0.550	0.610	1.500	2.000	2.220
Node No.	1	- 6	16	20	21
Time (sec)			Temperature	<u></u>	
.05	4720	70	70	70	70
.10	5130	70	70	70	70
.20	5450	73	70	70	70
.50	5782	100	70	70	70
1.0	5965	175	70	76	70
2.2	6081	348	77	70	70
5.2	6133	603	146	70	70
10.2	6149	857	297	71	70
20.2	6168	1230	565	78	73
30.2	6182	1522	790	97	86
40.2	6194	1762	983 .	125	108
50.2	6204	1966	1151	160	138
59.2	6212	2124	1285	197	170

Case B-1
Throat Location: 2.300" diameter

Case B-2
Throat Location: 2.300" diameter

Location Radius Node No.	Surface 1.150 1	Near Rear Graphite 2.185 15	Wear Rear Carbon 2.685 19	Near Outside (in steel) 3.0225 21
Time (sec)		Tempera	ture. *F	
.05	1880	70	70	70
. 10	2410	70	70	70
. 20	3000	70	70	70
.50	3778	70	70	70
1.0	4330	74	70	70
2.3	4900	132	70	70
5.3	5321	370	71	70
10.3	5579	774	82	71
20.3	5797	1413	143	91
30.3	5909	1881	235	142
40.3	5981	2240	343	216
50.3	6032	2524	456	304
59.3	6066	2732	559	389

Case B-3
Throat Location: 2.300" diameter

Location	Surface	Rear PG	Rear Graphite	Rear Carbon	Near Outside (in steel)
Redius Mode No.	1.150 1	1.195 6	2.000 12	2.750 20	3.000 21
Time (sec)			Temperature	تئے۔	
.05	4550	70	70	70	70
. 10	4980	71	70	70	70
.20	5330	81	70	70	70
.50	5692	155	70	70	70
1.0	5881	307	71	70	70
2.3	5995	592	96	70	70
5.3	6035	960	240	70	70
10.3	6065	1363	496	70	70
20.3	6105	1939	923	72	71
30.3	6135	2359	1266	80	76
40.5	6158	2690	1556	97	88
50.5	6175	2952	1797	122	108
60.5	6191	3169	2005	154	136
70.5	6203	3353	2187	193	170
80.5	6214	3511	2347	236	209
90.5	6224	3648	2489	282	252
100.5	6232	3767	2616	331	298
110.5	6239	3873	2730	383	346
120.5	6246	3967	2833	435	396
130.5	6252	4051	2927	487	447

Case 3-4
Throat Location: 2.300" diameter

Location	Surface	Rear PG	Rear Graphite	Rear Carbon	Near Outside (in steel)
Redius Node No.	1.150	1.195 6	2.250 14	2.750 20	3.000 21
Time (sec)			Imperature	-3	
.05	4550	70	70	70	70
.10	4980	71	70	70	70
.20	5330	80	70	70	70
.50	5692	155	70	70	70
1.0	5881	307	70	70	70
2.3	5995	591	. 78	70	70
5.3	6035	949	158	70	70
10.3	6061	1309	336	71	70
20.3	6097	1810	663	80	75
30.3	6123	2181	939	103	90
31.3	6125	2213	965	106	93

Case B-7
Throat Location: 2.300" diameter

Location	Surface	Rear PG	Rear Graphite	Rear Carbon	Near Outside (in steel)
Radius	1.150	1.195	2.000	3.125	3.375
stode No.	1	6	12	20	21
.ime (sec)			Imperature	ال	
.05	4550	70	70	70	70
.10	4980	71	70	70	70
.20	5330	80	70	70	70
. 50	5694	155	70	70	70
1.0	5882	307	71	70	70
2.2	5993	581	94	70	70
5.2	6035	954	237	70	70
10.2	6064	1359	493	70	70
20.2	6105	1937	921	70	70
30.2	61.34	2357	1265	71	70
40.2	6157	2683	1550	73	72
50.2	6175	2947	1793	78	75
60.2	6190	3165	2002	85	81
70.2	6203	3350	2185	96	90
80.2	6214	3509	2348	111	102
90.2	6224	3647	2492	128	120
99.2	6231	3757	2610	147	135

Case C-1
Entrance Location: 1.840" diameter (1.100" throat)

Location	Surface	Rear PG	Rear Carbon	Wear Outside (in steel)		
Redius	0.920	1.750	2.250	2.5375		
Node No.	1	5	9	11		
	•	•	•	**		
Time (sec)		Temperature, or				
. 05	620	70	70	70		
. 10	850	70	70	70		
. 20	1150	71	70	70		
. 50	1650	92	70	70		
1.0	2095	170	70	70		
2.2	2692	417	70	70		
5.2	3491	975	71	70		
10.2	4207	1675	80	72		
20.2	4925	2607	140	103		
30.2	5299	3214	243	177		
40.2	5528	3641	371	281		
50.2	5683	3955	507	399		
59.2	5783	4170	631	511		

Case C-2
Entrance Location: 1.840" diameter (1.100" throat)

location	Surface	Rear PG	lear Carbon	Near Outside (in steel)
Redius	0.920	1.750	2.625	2.925
Node No.	1	5	10	12
Time (sec)		Ţœ	perature. '7	
.05	620	70	70	70
. 10	850	70	70	70
.20	1150	71	70	70
.50	1650	92	70	70
1.0	2095	171	70	70
2.2	2692	418	70	70
5.2	3492	979	70	70
10.2	4210	1681	70	70
20.2	4928	2613	74	71
30.2	5301	3222	89	80
40.2	5532	3655	118	101
50.2	5689	3979	160	135
59.2	5791	4206	209	175

Case D-1
Entrance Location: 3.540" diameter (2.300" throat)

			· ·	- ·
Location	Surface	Rear PG	Rear Carbon	Near Outside (in steel)
Redius	1.770	2.625	3.250	3.5375
Node No.	1	6	12	14
Time (sec)		Inn	erature. °T	
.05	670	70	70	70 ·
.10	900	70	70	70
.20	1180	71	70	70
. 50	1690	90	70	70
1.0	2181	175	70	70
2.2	2842	445	70	70
5.2	3703	1054	70	70
10.2	4442	1810	72	70
20.2	5148	2799	99	82
30.2	5500	3433	162	123
40.2	5712	3876	255	194
50.2	5852	4200	365	285
60.2	5950	4445	484	391
70.2	6022	4635	605	501
80.2	6077	4783	725	612
90.2	6118	4902	841	723
99.2	6148	4990	943	820

Case D-2
Entrance Location: 3.540" diameter (2.300" throat)

Location	Surface	Rear PG	Rear Carbon	Near Outside (in steel)
Redius	1.770	2.625	3.625	3.925
Node No.	1	6	13	15
Time (sec)		Tong	erature. *F	
.05	680	70	70	70
.10	900	70	70	70
. 20	1180	71	70	70
.50	1690	90	70	70
1.0	2181	176	70	70 .
2.2	2842	446	70	70
5.2	3704	1059	70	70
10.2	4444	1817	70	70
20.3	5156	2815	71	70
30.3	5506	3446	78	74
40.3	5716	3880	95	85
50.3	5055	4212	124	107
60.3	5954	4461	164	139
70.3	6027	4655	214	182
77.4	6067	4768	254	216

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APPENDIX B

Calculated Stresses in Free-Standing Cylindrical
Shells of Pyrolytic Graphite

NOTE: See Appendix F for definitions of symbols used in this Appendix.

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APPENDIX B

PHYSICAL PROPERTIES INPUT DATA

Pyrolytic Graphite:*	Young's Modulus	Ea	=	4.5×10^6 psi
٠.	Young's Modulus	Ec	.=	1.5×10^6 psi
	Poisson's Ratio	vac	•	0.90
	Poisson's Ratio	v _c a	=	0.30
	Poisson's Ratio	-		-0.16
	Shear Modulus	•		0.2×10^6 psi
	Shear Modulus	G _c	=	2.68×10^6 psi
	Total Thermal Expansion	$\alpha_{\mathbf{a}}^{} \Delta \mathbf{T}$	=	-0.00144 in/in
	Total Thermal Expansion	$\alpha_c^{\Delta T}$.=	-0.0432 in/in

^{*} Reference 7.

CASE NO. 1 E1 •45000000 07	E2 .15000000 07	NUI •3000000• 00	• 00 NUZ •90000000•	00	NU316000000
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APPENDIX C

Calculated Stresses in Composite Cylinders with

Pyrolytic Graphite Coating on the Inside

NOTE: See Appendix F for definitions of symbols used in this Appendix.

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APPENDIX C

PHYSICAL PROPERTIES INPUT DATA

Pyrolytic Graphite:*	Young's Modulus	E	-	4.5×10^6 psi
	Young's Modulus	Ec	=	1.5×10^6 psi
	Poisson's Ratio	vac	=	0.90
	Poisson's Ratio	v _{ca}) = (0.30
	Poisson's Ratio	v _{aa}	=	-0.16
	Shear Modulus			0.2×10^6 psi
	Shear Modulus	$^{\mathbf{G}}_{\mathbf{c}}$	=	2.68×10^6 psi
	Total Thermal Expansion	$\alpha_a \Delta T$	•	-0.00144 in/in
	Total Thermal Expansion	$\alpha_c^{\Delta T}$	=	-0.0432 in/in
Molded Graphite:**	Young's Modulus	E	(=)	1.5 × 10 ⁶ psi
	Poisson's Ratio	υ	=	0.35
	Total Thermal Expansion	$\alpha_{\mathbf{a}}\Delta\mathbf{T}$	=	-0.0046 in/in
	Total Thermal Expansion	$\alpha_c^{\Delta T}$	=	-0.0070 in/in
Extruded Graphite:**	Young's Modulus	E	=	1.5×10^6 psi
	Poisson's Ratio	υ	-	0.35
	Total Thermal Expansion	$\alpha_a \Delta T$	•	-0.0044 in/in
	Total Thermal Expansion	$\alpha_c \Delta T$	=	-0.0076 in/in

^{*} Reference 7.

^{**} Reference 9.

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. 1	1000000, 07 E2 .15000000, 07 NU1 .30000000, 00 NU2 .90000000, 00 NU316000000, 00	85714+. 07. 62-+20000000+.06- AA1-+14400000+02-AA2-+43200000+01	1000000, 00 RO .59000000, 00 E .15000000, 07 NU .35000000, 00	0000000-02 GTHEIA46000000-026270000000-02RG +9900000000	1076036. 07 C12 .21774193. 07 C13 .95046082. 06 C22 .28064516. 07 C44 .20000000.	51612 • 06 B2-12750966 • 06	TRAIN6311549602	R SIGMA R SIGMA THETA SIGMA Z U R	• 00 • 30000000 - 016496	. 0049700000, 0120967400, 0421590970, 05	• 0011140000 0225415000 0421525360 05	• 0018440000 0229839200 0421461140 05	• -00	• 0036440000 0238619200 0421336870 05	0047110000, 0242975400, 0421276770, 05	0000 0071680000 0251621200 0421160550 051902000502	0085550000 0255911100 0421104390 05	-00	11641000.03	•	• 0015136000 + 0372858300 + 0420892460 + 05 -	. 0017030000. 0377042700. 04 -	• 0019026000 • 0381207200 • 0420793890 • 0522984820 •-	5-00	•	+ 0025580000+ 0393579300+ 0420654930+ 05	• 0027953000 · 03 -	0000, 0030415000, 0310172910, 0520568050, 0525789631, 02
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> -.30420774. 03 -.19827329. 03 -.12439162. 03 -.70824666. 02 -.30753407. 02

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		.86012000.	• 05	1.115.0702
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	8208000	25244590	• 05	1676439502
	6061000	36233400	• 05	16299581,-02
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	•	.23146818,	.14181489, 04	30352924 02
		•16988296•	.14161489. O4	37424955
•	13148.	.13420318.	.14181489, 04	4443768302
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575000		١	- 70	20587480	02	.43158058
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1622000	30630000	•	1	20537100	6	70-1618174
11620000	10520509 •		i	20212360	6	******
116555000	60450000	00186/8/90	i	.046/8407	6	١.
1650000-0	10105000			101000000	5 8	
6800000	-11169990	0365024500		20416440	600	4710557402
.000569	12259600.	i	١	20393200.	9	3
710000.		i	1	20370250.	60	48229423+-02
11725000. 0		0 0	•- 50	20347590.	9	
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770000	-18058700	1	•	-20281330-	50	
•11785000• 01	19286000.	399379	١	20259810	02	5103144002
•11800000• 01	20539000-	0310141040+	05	20238580+	92	515905704-02
4	SIGNA R	\$16MA THETA		1644		-
.118000000 01	20541059.	.72355	03	12959155.	8	5159057702
2600000.	14830137,	03 .66644292.	ĺ	12959154	8	55735864
•13400000• 01	01111	1	03	2959154.	ð	59843754,-02
4200000	-	.57981	į	29591544	\$	20568
•000000	28371037	•	03	2959154.	8	6797127902
-10-400000861			3	1	\$	- TOWNSOCHART

CASE NO. 8	1	;	and the second	To the second se
E1 .45000000, 07	£2 .15000000 07	NUI .30000000. 00	0 NUZ .90000000.	00 NU316000000, 00
61 .26785714 07	62 .20000000 06	AA11440000002	AA243200000	01
RI .11500000 01	RO .12000000. 01	E .15000000 07	NU .35000000 00	
GR4600000002	GTHETA46000000	-02 62 70000000	02 RG -16000000	• 01
C11 .63076036, 07	7 C12 .21774193. 07	C13 •95046082•	06 C22 .28064516•	• 07 C44-•20000000 0
8110451612.06	8212750966, 06		9	;
AXIAL STRAIN-	5850798602			
œ	SIGMA R	SIGMA THETA	S I GMA - Z	a 5
.11500000.01	. 300000000.	32517500.	19328290. 05	32773228,-02
.00052	•		90	33739601 02
50000	•	•	. 05	
•11575000• 01	-24580000 02	43405600	4	
25000.	20 100001375-	50588400+ 04	- 19079390 05 -	4 300 3021 3 4-02
50000			000	38550321
15000.	•		50	.39508325
1000001	•	61251700, 04	18940790, 05	.40465029.
725000		-	1	** 1420353 v - 02
150000		•	18852720, 05	.42374386,-02
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8C00008	•	•	90	
825000		•	02	452287681-02
11820000911	-1/30/000• 03	82190400, 04	_	•
•000006	34000	89058700 -	18608460- 05	4807144002
925000.	10		•18570570 • 05	49017090
.00003	4318000.	3100.	0	
.11975000 01	304000	0300	05	*50904000 - 02
.12000000.01	28352000. 03	10263430, 05		
~	SIGMA R	SIGMA THETA	SIGMA Z	2
.12.000000, 01	28355122, 03	.10126941, 04	1.00	-
2800000	•	•	70	.56206222.
3600000	140r2233, 03	•	70	6051526502
4400000	•	•		•64781418•-02
500000	93831114	9	-	20114751-02
•16000000001	-2444444 02	.72914040, 03	-19790021. 04 -	7321078602

NU316000000 00		01	07 - C44 - 20000000+ - 04	Marie a marie		4 11	22357025 02		25531265,-02	ò	•	•		-	345519411 02	8074537.	39626202 02-				334550	48866610+-02	50395600 02	51921590 02	3444580 • - 02	a D	34446024-02	5802621902	•62546213 • - 02	6791464302	1439493	5827161 0-02
00 .00000000	00 00	.16300000.	-28064516-		† h		05 2	5	05 2	i	05 -	0.2	•		050	05		4.1	2	2	5 -	05 = 4	05 5	. 055	05 5		1	64 5	i	i	•	7 40
	NU -35000000 00	02 RG .16	06 622 -28	1		S IGMA 7	17657430	17559970.	17465060	17372650		-17195170	17110030	-17027230	-16868510	16792520.	-16718720v	-1664/090	-16510170	16444820.	16381500,	16320170	16260809.	16203380.	16147850,	SIGMA 2	-27995370	.27995370.	.27995370.	.27995370.	7995	•27995370•
NUI -3000000 00	E .15000000 07	62 70000000	C13 -95046082			SIGNA THE	.55680000 03	47360000, 02	•	•	•		•	- 4740 / 300 0 04			58688800 04-			•	86403000. 04	91842100 - 04	•	•	10796090. 05	SIGMA THETA	-12428594v 04	•	•	•	•	.90187636, 03
E2 .15000000 07	0.12300000.01	GTHETA4600000002	-C1221774193+-07	B212750966+ 06	5344079902	SIGNA R	.1000000001	.89000000 00		100000	•		25280600 02		64210000 02		1195320000 02	-11452000 03	_	•	21733000, 03	24587000 03	27602000, 03	•	34096000 03	SIGMA R		•	•	10350548, 03	496294	-1461461403
E1 -4500000, 07	.11500000	GR46000000-02	C1163076036+-07	8110451612- 06	AXIAL STRAIN	R	.11500000, 01	2400000	•	•	•	700000	11 740000 01		860000	•000006	11980000-01		000000	2100000		180000	2220000.	2260000	.12300000 01	œ	•12300000 · O1	•131000000 01	3900006	1000001	15500000 01	300000

CASE NO.	10									
E1 .45000000	0. 07	E2 .15000000.		07	NUI .30000000.	000 • 000	NU2	.90000000.	00 NU316000000	00
61 .26785714	4. 07	62 .20000000	0000 00		AA114400000,-02	0000	AA2-	•43200000	001	
RI .11500000.	0, 01	RO .11800000.	0000 01	1 E	.15000000.	0. 07	NU .35000000.	F	00	
GR4600000007	20-00	GTHETA4	ETA4600000002	0	25	70000000	02 RG	.18800000.	000• 01	
C11 .63076036.	36. 07	C12	.21774193. 07	07	-C13 .950	.95046082.	06 C22	.28064516.	\$16 • 07 C44 •20000000	0 •
8110451612.	2. 06	8212750966•	90 •9960	9						
AXIALSTRAIN		6584302502	-20-0							
œ		SIGMA	MA R		SIGMA TH	THETA	SIGMA	7	2	
.11500000	01	•2000	- 00	10	0	0.04	0	• 05	N	
	ن ا	78500000		10	61527600		22171960.		41385451,-02	
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.11680000	0:0	11086000		60	84363300		21899320	. 05	•	
•11695000•				60	86409000		21876310	60	.48109623	
11725000	10) 26 T •	14399000	5 6		•	21853610	5	4866708302	
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.11755000	10	1672		60	94543000	•	21767180	0.0	.50336850	
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•11800000•	01	20381900	10001	03	10059280	0 0 05	21723300	90	52002810,-02	
œ		SIGMA	MA R		SIGMA TH	THETA	SIGMA	7	2 3	
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.14600000	01	87198481	•	02	.35220016	•	.71629681	03	65875046	
• 16000000	010	50433148		02	.31543483	3 03	.71629681	03	7272125302	
• 18800000		37037037		70-	-26500172		-71629630	0 0		
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0000. 00 NU316000000. 0	0000 01	00 •0	.19000000, 01	.28064516, 07 C44 .20000000	20 1 20 1 20		x	053353395002	05 34489791 02	35444233	563615695	37348957	053924H19102	40195793	41142084	-45081046	43030728	43973130	i	45854144	46792786	05 - 6844410-02	49601430	50535260	5	52399390	2	045239940902	045962353402	66748370	-	048774662102
00 NUZ .90000000	02 AA24320000001	NU .35000000.	02 RG	06 - C22	;		SIGMA 2	0	21430810.	21380810.	21431420-	21283510	-021230190	21144160.	21099470.		21012580.	20970390.	20929020-	20888470.	20848720.	- 20771420	-120734250-	20697650	20661820.	20626750.	SIGMA 2	.11383633.	.11383633	_	.11383633.	11383633
NU1 -30000000 (AA11440000002	E .15000000 07	02 62 70000000	C13 .95046082.			SIGMA THETA	₩	35643800, 04	3700.		•	53394200 04			e3868900 · O4	67332000, 04	•	•	•	•	= a7a20100 04				10121080. 05	SIGMA THETA	.65064729, 03	•	•	•	•40175329•03 •37106307•03
E2 .15000000 07	62 .20000000 06	RO .12000000 01	GTHETA46000000	C12 .21774193. 07	R212750966. 06	6327673002	SIGMA R	.2000000001	731000000 01	•		33770000 02	550400000-02	66730000	•	1	•	•	•	C	•	- 20246000 - 03		3977000 0	25934000 03		SIGMA R	•	•	6.0	9674333.	
111	4. 07	0.01	002	36. 07	2. 06	2		01	01	01	01	6.0	5 5	010	01	01-	01	01	0	6	2 2		5 5	10	01	01		01	01	5	2 3	01.
CASE NO. 1	61 .26785714	RI .11500000.	GR46000000	C11 .63076036	8110451612	AXIAL STRAIN	œ	.11500000	.11525000.			•11600000	11650000	.11675000	.11700000		_	~	┥,	_		11900000	:=		.11975000	.12000000.	α	.12000000	.13400000	.14800000	•16200000	.17600000.

CASE NO. 12				
E1 .45000000 07	E2 .15000000 07	NU1 .30000000. 00	NUZ .90000000 00	NU316000000 00
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RI .11500000 01	RO .12300000. 01	E .15000000 07	NU .35000000 00	
GR46000000-02	GTHETA46000000-	-02 GZ70000000	-02 RG .19300000 0	1 ·
C11 .63076036. 07	7 (12 -21774193+ 07	C13 .95046082.	06 C22 .28064516+ 07	7 C44 .200000000 06
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AXIAL STRAIN	597615350-02			
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11500000 01	-• 00		90	23406206 02
	•10800000 01	•	• 05	24972942.02
	ı	•	- 62	1
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.116600000 01	_	•	60	29649578+-02
		•	1 22	
	23/00000+ 02	34685200 04	050	34292195 02
11820000	= 4702000 = 02		60	3583246002
860000	- 1		100	373692060-02
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020000	•	•	. 05	45005083-02
•	•	_	9319380, 05	
2100000	8430C00 0		19252850. 05 -	. :
21400000	50 +0000017*-		19192440 05	49543130 02
9171			19133960. 05	51050710 02
12 250000		-10026910	. 05 5	2555330 02
2300000	•	10551560.0	19022690. 055	4057000 02
ď	C I GWA D	SIGMA THETA	SIGMA Z	2
10 .000000000	331407B	-	04	54057025 02
		67650874	70	.6154434602
15100000 01	14362903	.59696103. 0	8	۰
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.0000061	6843		1	00627731
10 .000000661.	5925925903	•45333229• 0	•16944358 · 04 -•7	•

E1 .450000000	0. 07	E2	.15000000.	01	NO1	•30000000	00 •0	NU2 -9000	• 000000006 •	00	NU316000000.	00
61 .26785714	4. 07	62	.20000000	90	AA1-	AA114400000,-02	002	AA24320000001	00000	-01		}
RI .56000000.	00 •0	8	.59000000	8	E -15	-15000000.	10	NU .35000000.	00 • 00			
GR 7600000002	002	GT	GTHETA4400000002	• 000		624400000002		RG	.99000000	00 • 00	. (
C11 .630.76036+	03 96	- C12	12 -21774193+	3+ 07	4 (13	3-95046082-06	582.	553	-28064516+	16+ 07	- C++ -29000000	90 + 00
811045161206	2 06	- 85	821275096606	90								
AXIAL STRAIN		3	0238823+-02					iei				
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56150000•	0	1	.52400000	• 01	• 1	17097000.	10	-111996310	- 60-4	83	•83331910*-03	
56300000	ç		.91400000	•	•1.	.12250600.	. 40		• 05	894	.89499620,-03	
56600000	600		13040000	950	2	74-30-2000	69	-11657910	9 6	201	956547304-03	
	00		-13060000		2	.21341000.	030	-11581570	-05-	101		
• 26900000	Ç		.11830000	•	190-	.68785000.	03	11506770	• 05	114		
.57050000.	00		.93800000	-		.11597800.	• • • • • • • • • • • • • • • • • • • •	11433470	- 60	120	120154561-02-	
	000		• 56 900000	5		.16292800.	40	11361670		126		
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5	00		20920000		-3	34831600.		11069000	200	-150		
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• 20 000 to 10 0	3 6					•00404610•	3 6	-10/17/01-	_		18659696 - 02	
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• 28000000	00		12821651	• 03		.19250420+	70	•H930654	8	198	54110+-02	
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.83000000	o c		12269527	• 03		19282714.	03	.53963036.		388	:	
•00000016•	200		-14374925	9	Þ	64785829	\$	-9696969	1	1	400000	

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00 RO .61 -02 GTHETA 00 B212 06 B212	21774193 07 750966 06 750966 06 750960 02 1640000 02 160000 02 160000 02 160000 03 1264000 03 1264000 03	Ct3 - Ct3 - Ct3 - Ct3 - Ct3 - Ct01 - 5571 -	-350 2 RG 2 RG 11737 11150 11120 11120 110838	01 · 07 · 03 · 03 · 03 · 03 · 03 · 03 · 03
-02 GTHETA -07 -C12 - 06 8212 06 8212	447000000- 21774193 07 750966 06 40802 1640000 02 1660000 02 1660000 02 1660000 02 1660000 03 1264000 03 1264000 03	C13 - C13 - C13 - C13 - C13 - C13 - C10 -	516 11737 111575 111120 110977 10777	01
06 B2-12 06 B2-12	21774193 • 07 750966 • 06 108 • 02 1644 R 51644 R 5164000 • 02 5680000 • 02 5680000 • 02 786000 • 03 786000 • 03 786000 • 03	S S S S S S S S S S S S S S S S S S S	516 111737 111575 111507 111207 11120 11120 11120 11120	13912 24468 34985 45464 55907 76684
37906	•		SIGMA Z 11737170• 11575800• 11419240• 1120800• 10977910• 10838950•	1391243003 2446809003 3498526003 5590719003 6631975003 7668481003
2.5.6.6.6.6.6.6.6.6.6.6.6.6.6.6.6.6.6.6.		<	SiGMA 2 11737170• 11575800• 11419240• 1120806• 10977910• 10838950•	13912430,-03 24468090,-03 34985260,-03 55907190,-03 66313750,-03 76684810,-03
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	Land Land		11575800+-11419240+ 11267370+ 11120080; 10977310+- 10838950+	2446809003 3498526003 5590719003 6631975003 7668481003
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. 6.			11120080; 10977910; 10838950; 10704900;	55907190;-03 55907190;-03 6684810;-03 87021180;-03
•			10977910+ 10838950+ 10704900+	55907190-03 56319750:-03 76684810:-03
11.			10838950	7668481003 87021180:-03
		•	L	8702118003
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•1	2036000 03	23375200, 04		13820790,-02
7.	1	•	99865400 .04	
6.	3380000 02	38397900. 04	98803800, 04	1584642302
4.	3700001	•	L	-
·•	*430000	•	-	•
m •	0620000	•		18863032 02
	63400000 01	6//18600.04	94916800.	
\$ • •			93179400 04	2185494102
5	SIGMA R	SIGMA THETA	SIGMA Z	2
•	7433296. 02	4		
٠.		•	•	28416832 02
•		.25122092 03	.10533543 5-04	34748259,-02
•	•	71518,	.86411107, 03	40926517,-02
00	7069814 v 02	-7H48818+ 03	**************************************	**************************************

CASE NO. 1	15				
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61 .26785714	•• 07	62 .20000000 06	AA11440000002	2 AA24320000001	01
RI .56000000.	00 •0	RO .64006900. 00	E .15000000 07	NU .35000000 00	0
GR 7600000	2002	GTHETA44000000-02	6244000000	02 RG -10400000	. 00
C11 •63076036•	10 496	7- C12 •21774193+ 07-		-06C22-*28064516*-07	16+-07
81-,10451612, 06-	90 • 3	- 8212750966. 06 -			4
AXIAL-STRAM	Ţ	3492598405			
œ		SIGNA R	SIGNA THETA	SIGNA 2	- A D
• 26000000	00		.14023990, 05	11480520, 05	.71021620,-03
.56400000	90			1	.53594410=-03
.56800000	င္ပ	191000.			.36277590,-03
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.59200000.	00				65494400,-03
· 59600000	8	1		П	65130920-03
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• 00000000	000			_	13154076 02
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.628conoo.	9				21237694 02
•63200000	00	380000	100		
•63600000		641000+ 0		1	-1244236771-02
• 640000000	00	•15291000 • 03	10481280. 05	14220600, 04	2600737902
α		SIGMA R	SIGMA THETA	SIGMA 2	& D
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.11605000.			54861790. 04	10849710, 05	352377981-02
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.11650000.	01	-64760000 02	61612700.04	10761790. 05	3703373802
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.11800000	0	15640311+ 03	. 14265596v 04	.98611740+ 03	
.12600000.		70358925 02	.98169251. 03	.66252940. 03	4952276202
.13400000	10	-19376259+ 02	.59390310 . 03	.74467507 · 03	
.14200000		.53547777. 01	.25205698, 03	.63365674. 03	6221111702
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			.98462300*	-364752799-02
	10760000, 03			374773649-02
			\$1001;	384766709-02
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	9852000, 0	.84718600. 0	.95296600.	4346120002
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61 .2678571	4. 07	62 .20000000 06	AA114400000-02	2 AA243200000	101
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1590	01	.0000665	•	•	3185003002
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62000	CI.	.36910700.	•		3305089002
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66500	0.0	-55370000	51607000 04	11608390* 05-	34244044-102 348483461-103
00099	01	.62070000		11548450	.35446309
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7000	01	10788500. 03	•	11376520. 0	39624127 02
8500	0 1	1643	006	t1349000 · 05-	39618667 s-02
•11800000•	01	12524000. 03	71566090 04	11321790, 05	4021274002
		SIGMA R	SIGMA THETA	SIGMA 2	2
w.	01	1.0	•	-	40212794,-02
3	01	•	•	•	51585880,-02
4	01	•	•	8	23
160000000	0	.94899407, 02	12860459, 03	. • 31009018 • 03	7328587702
.18800000	010		1605577.	5	94207100,-02

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1750500) C	-02202500		90934676
11725000.		02900004-0	32981490	-100	-30082363
11750000		C	36745000.	2693.	31089554.
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1800000		54650000+ 02	44225600,	11058210, 05	•
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.11540090.	0	2920000.	•34000400•	11345470,-05	12583780,-02
.11580000.	10	3530000	.27629200.	•	1424558502
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11700000	10		60 -00008610- 04	-10916850 05	
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•12200000	5 6	1263000. 0	74343300	7259700, 0	872300,-
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.15100000.		7662222	.40311874.	0035765.	•66191502
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AFRPL-TR-65-57

APPENDIX D

Calculated Stresses in Composite Cylinders with

Pyrolytic Graphite Coating on the Outside

NOTE: See Appendix F for definitions of symbols used in this Appendix.

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APPENDIX D

PHYSICAL PROPERTIES INPUT DATA

Pyrolytic Graphite:*	Young's Modulus	Ea	=	$4.5 \times 10^{\circ}$ psi
	Young's Modulus	Ec	=	1.5×10^6 psi
	Poisson's Ratio	v _{ac}	=	0.90
	Poisson's Ratio	v _{ca}	=	0.30
	Poisson's Ratio	vaa	=	-0.16
	Shear Modulus	G	=	0.2×10^6 psi
	Shear Modulus	Gc	=	2.68×10^6 psi
	Total Thermal Expansion	$\alpha_{\mathbf{a}}^{}\Delta\mathbf{T}$	=	-0.00144 in/in
	Total Thermal Expansion	$\alpha_c^{\Delta T}$	=	-0.0432 in/in
Molded Graphite:**	Young's Modulus	E	=	1.5 × 10 ⁶ psi
	Poisson's Ratio	υ	=	0.35
	Total Thermal Expansion	$\alpha_{\mathbf{a}}\Delta \mathbf{T}$	=	-0.0046 in/in
	Total Thermal Expansion	$\alpha_c^{\Delta T}$	=	-0.0070 in/in

^{*} Reference 7.

^{**} Reference 9.

00 NU316000000 00	-01		0.01	6. 07 C44 .20000000.			, 'S	8743916002	. !	8629101002	8571651002	8514174002	84566680,-02	8399135002	1 - 0 3 4 1 3 7 0 0 0 - 1 0 2 1 0 3 1 0 1 0 2 1 0 3 1 0 1 0 3 1 0	-82263600-02	8168721002	81110460,-02	8053341002	7995610002	79378490 02	78800550 02	70-0046777	7706505002	76/8595002	8	2		- 127086081-02 - 7448714303	7346140502	72229125 02	7099010002	20-10010101 - VOIT - VO
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•	01	•32062000	•	•	•	6305263002
•	5	.34446000	•	•	0526000.	61456750,-02
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	01	.30982148	•	•	•	•
	010	.20986703	•	•	•	44161280,-02
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90 8 C44 .200000000 NU3--16000000 --16353793--01 --16238273--01 --16180490--01 --16007059,-01 --15949230,-01 --15891373,-01 --15717725,-01 --15601889,-01 --15543943--01 --15370034,-01 --15189293,-01 --15066212,-01 --14942815,-01 --16469246--01 --164115319-01 --16296035,-01 --16122696.-01 --16064889.-01 --15833502 -- 01 --15775613,-01 --15659812,-01 --15428017--01 --15312040--01 --15312062--01 --14819095 --01 --15485986,-0] --14695049.-01 œ **5** ⊃ C22 .28064516. 07 0 NU2 .90000000 00 AA2--43200000 -01 .31000000 NU .35000000 00 44444444 400 4444 444444 3000 .26297825. .26206657. .26114906. SIGMA 2 -.85120300. -.85180600. -.85363700. -.85676300. -.85868300. .26478443, .26388417. .26022564, -.85302300 -.85425500 -.85487600. -.85803900 -.65998200. -.86329700. SIGMA Z --85241300 -.85550200 -.65613100. -.85740000 -.85933100. -.86063800 -.86129600, -.86195900 -.86262600, -.86397100. RG 62--46000000-02 C13 .95046082. 06 NU1 .30000000 00 AA1--14400000,-02 00000 E .15000000 07 90 400 9 90 9 90 300 9 9 10 40 SIGMA THETA SIGMA THETA --15102800. .42991022. .42962229. .43002807. -.15025750. --14948630. --14871420. --14794180. --14716850. -.14639470. --14484510. --14406950, --14329300. --14251580. -.14173790. -.14095970. --14018070. --13940110. -.13862050. --13783950. --13705790, --13627560. --13549270. .42966785. .42973066. .42981122, --14562040. GTHETA-.70000000.-02 C12 .21774193. 07 02 020 02 020 02 020 02 60 03 03 .27557777 02 .50000000-01 .20000000. 06 .1500000000 .32000000 01 90 .70400000. .27870000 .81615925. .54761851. .20970000 .10812740. .14010000. .34760000. .416100CO. .55220000. .61980000. .66700000 .75420000. .82100c00. .95340000. .10191000. .11500000. .13430444. .48410000. .88730000 .10847000. .12148000. .12794000. .13437000. 82--12750966. SIGMA R SIGMA R --38685602+-02 E2 2 62 C11 .63076036. 07 0 90 0 5 GR-.46000000.-02 0 0 0 0 0 16 01 0 6 \overline{c} 0 60 0 5 5 0 0 5 0 222 000 01 .45000000 .26785714. .32300000. B1-.1C451612. AXIAL STRAIN •31830non• .3163n000. .32150000. .3209000C. .32015000. .32000000 .314Cn000. .31200000 .32195000. .3218c000. .32135000. .3212000c. .32105000. .32075000. .32060000. .32045000. .32030000 .32000000 .31000000 .32300000 .32285000. .12270000. .32255000. •3224C000• .32225000. .32210000. .32165000. œ œ

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APPENDIX E

CALCULATED STRESSES FOR VARIOUS TYPES OF SUBSTRATE GRAPHITE

MOTE: See Appendix F for definitions of symbols used in this Appendix.

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APPENDIX E

PHYSICAL PROPERTIES INPUT DATA

Pyrolytic Graphite:*	Young's Modulus	E	=	4.5×10^6 psi
	Young's Modulus	Ec	=	1.5×10^6 psi
	Poisson's Ratio	vac	=	0.90
	Poisson's Ratio	v ca	=	0.30
	Poisson's Ratio	v _{aa}	=	-0.16
	Shear Modulus	G	=	0.2×10^6 psi
	Shear Modulus	G _c	=	2.68×10^6 psi
	Total Thermal Expansion	$\alpha_a \Delta T$	7	-0.0049 in/in
	Total Thermal Expansion	$\alpha_c \Delta T$	=	-0.0500 in/in
Molded Substrate (ATJ):***	Young's Modulus	E	=	1.18×10^6 psi
(A13).	Poisson's Ratio	υ	=	0.35
	Total Thermal Expansion	$\alpha_a \Delta T$	=	-0.00772 in/in
	Total Thermal Expansion	$\alpha_c^{\Delta T}$	*	-0.01029 in/in
High Density	Young's Modulus	E	# **	2.09×10^6 psi
Substrate (ZTA):**	Poisson's Ratio	υ	()	0.35
	Poisson's Ratio Total Thermal Expansion			- 88
		$\alpha_a^{\Delta T}$) =	0.35

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APPENDIX E (continued)

Molded Fibrous Substrate (PT-0114): *** Young's Modulus $E = 0.30 \times 10^6$ psi Poisson's Ratio v = 0.35Total Thermal Expansion $\alpha_a \Delta T = -0.00528$ in/in Total Thermal Expansion $\alpha_c \Delta T = -0.00714$ in/in Extruded Substrate (Speer 580): v = 0.35Total Thermal Expansion $\alpha_a \Delta T = -0.00714$ in/in Total Thermal Expansion $\alpha_a \Delta T = -0.0005$ in/in

Total Thermal Expansion $\alpha_c \Delta T = -0.008$ in/in

^{*} References 7 and 8.

^{**} Reference 11.

^{***} Reference 10.

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NU3--16000000, On GIHFIA-.77200000.-02 GZ-.10290000.-01 RG .12900000. 01 NU2 .900000000 00 AA1--49000000-02 .AA2--50000000-01 NI .35000000 00 NUL .300000000 00 E .11800000 07 E2 .150000000 07 .59POPPED 00 **90 • 000000002•** 0 25 F1 .45000000 07 GR-.7720000-02 G1 .26785714. C7 RI - . 56000000 00

CASE NO.

C44 .20000000 06 C22 .2d064516, 07 C13 .95046082. 08 C11 .63C76036. 07 C12 .21774193. 07 32-,16166128, 06 A] -. 14443547, OF

-.9842451C.-C2 AXIAL STRAIN

--3109500B--02 --31779276.-02 --32462293•-02 --33824049--02 --34504037-02 --35102235.-02 --35859252 --02 -.36>3>180.-02 --372099189-02 --37883546--02 --38556084.-02 -- 39227511 •-n2 --419026530-04 --43<34058--02 --43698256+-02 --44561474•-02 --44561509,-02 --55906830+-02 -.67141183.-02 -.78310713,-02 --89439095 --02 --33144061--02 --39897875--02 -- 4026718/--02 --41235455--02 --42568850--02 --10053973.-01 50 03 03 0000 05 5 05 05 50 05 05 05 05 90 .56710877. -.21486000, .56710877. .56710877, -.22343060. -.22154880. --22108170. --22033040. --21747760. --21424110. --21304440. --21028560. SIGMA Z .56710877. -.22263170. -.21959443, -.21667360. -.21616820. -.21660160· --21614010 549300. -.21363550. -.21246630. -.21190160, --21135000. -.21081140. .56710877. -.21 40 ć 40 03 03 .637850FC. C? 40 40 20 40 40 **7**C 40 4 40 .32206456, 03 SIGNA THETA SIGMA THETA .2296967. .14650485. .12582234. - 3464KOOO --- H347C000. --13203800, -.228409nC. -.27621900. -.32378700. -- 37110500. -.4181890C. -.51162700. -.60413800. -.65005100. -.69573200. -.74119200. -· 78643300. -. 83145200 · -.876253nO. .11143106, .14437000. --16035000. -046532400. -.5579980C. .17821015. ٤ ζ --2755CCSD+ C2 S ç S Š 20 C C Č -. 11826833, na -.66778821, n2 -.35173526. 02 -- 14391017, 02 .26222221 ,-03 -27.00000-11 In Trunche ri --21063323, -.7750000 - 046/5010r -.11500000 --13197000, --15004000 --18937000+ --21059000. • " -.363ncnn. --13120000 -.19/21-i -.3655GnCr. --70640nn. --8431000° • 50006766 • --16919000 --78(nonn. - 5581200C. SIGN'A R SIGVA R C C C ć C ć ç C ů. 55 .56250000 .115cnon. .562-0-0-\$67500Cr. .572 non. . F75. PRINC. \$79500CF 581: nncr. 8700000 •129000C0• . 560 CCC. £615ccc. . 4765000C. . F7ATACACA. .588500co. . 4900000° *2645rnn. ,57050rcc. .57450000 .5855000c. . 4907 COOL •1010000tol• • 2000000995 \$691 nnon. .58400000. .587:000G . 730000F1. ~

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E2 .15000000 07 NH .30000000 00 NH .90000000 00 NH -16000000 00 62 .20000000, 06 AA1-.49000000.-02 AA2-.50000000.-01 E1 .45000000 C7 G1 .26785714. 07

RI .56000000. On RC .61000000. On F .11800000. O7 NU .3500000. On

GR-.77200000.-02 GIHEIA-.77200000.-02 GZ-.10290000.-01 RG .13100000.01

C44 .20000000 06 C11 .63076036, 07 C12 .21774193, 07 C13 .95046062, 36 C22 .24064516, 97

RI-.14443547. 06 42-.16166128. 06

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-5675nnn. nr				21461600, 05	278224339-02
.5700 GGGG .		7 +30c06/5.	_	2131727b, 05	28979641 02
.57250C00. On		.85240000 C	2 .18295700, 04	21177450, 05	3013327002
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JC . NULL . 2 . 9.	•	• 66060000	2 82277300, 04		444344402
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.750cubub. un	_	958n3994. C2	2 .18922590 03	.89634438, 03	58478156,-32
		54494263, C2	2 .14791617, 03	•8963443H• 03	6981470202
			2 .12227381, 03	.89634438 03	81098957 02
		11850404. 02	.10527231.	.89634438. 03	92349692 02
•13100000 01		23308641,-02	.93424240.	•6963443B • 03	10357765,-01

E1 .45000000. 07 F2 .15000000. 07 NUL .30000000. 00 NUZ .90000000. 00 NU3-.16000000. 00 GR-.77200000.-02 GTHFTA-.77200000.-02 GZ-.10290000.-01 'RG .13400000. 01 61 .25785714, N7 62 .20FPC000, OK AA1-.49CPOOPP.-02 AA2-.5000POOP-01 RO .640(0000.00 E .11800000.07 NE .35000010.00 RT .56CDDDCD. DO

CASE NO.

C11 .63076F36. F7 C12 .21774193. 07 C13 .95P46P82. 06 C22 .28064516. 07 C44 .20C00000. 06

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--7C686427, 07
--4C683211; 07
--17980023, 02

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C44 .20000000 06 NU3--1600000), 00 --691549419-02 -- 70294211 9-02 -- 72567958 -- 02 -- 73705431 -- 02 -- 77096582 -- 02 -- 18224000 -- NZ --62723370--02 --87198890--02 --89428350 --02 --68014033--02 -.71431884.-02 -.74835355,-02 -.75966729 -- 79351730 -- 02 --60477003--02 --01600950--02 --63644360.-02 --84963930--02 --6831431C•-02 --90541040--02 --86082110+-02 C22 .28064516, 07 RG .19000000, OI NUZ .90000000 00 AA2-.50000000.-01 E .11800000, 07 NU .35000000, 00 05 25 05 05 CS 05 05 05 0.5 S 05 95 05 05 05 05 -.20282593. -.2n222503. --20163440. --20105409+ --20048370. -.19902340. -.15937290, -.19630120. -.19777983. -19726790 -.19350250. 51GF.A 2 --20343720+ -.19863220, 96/6540. -.19627210. --19578800. 9531310, --19484710+ -.19439010, -.19394190. -.19307170. SIGMA 2 67--10290000--01 -C13 .95046082 · 06 NU1 .30000000. 00 AA1-.49000000.-02 70 4 7 40 0 5 40 40 Č 50 **5** SIGMA THETA SIGMA THETA -.1309190C. -.1709650C, - 4 2 5 0 5 4 7 0 0 • -. 329454n0. -04077G000--.46529300. -.56224800. -.751908nc, -.7893R400. -.8638800. -.21084100, --60048900 -.63857400, --67650400. -.7142R200. --2900R500• -.36865900, -.82671100. -.50091900. -.446578CC -.52385000 GTHFTA-.7720000--02 C12 .21774194, 07 93 5. 2. 22 02 5 C C 6 6 ç 2 2 2 7 2 •10000000• \overline{c} E2 +15000000 07 62 .20000000 06 RO .12000000 01 A2-.16166128. 06 -.32600000 --19919000 --12001aco. --13440000 --16536000 --12350000 --18150000. -.2477cnco. -.6994cnon. -693470000 --10636000. -•14952nn• -.18192con, -.738conon. -.32210ngg. - 4046000r --.59340000. -.6132cncn. -.21715000, -.49490000. -.94673764.-02 SIG RA R SIGNA R (11 .63076C36 11) ٥ E1 .45000000 07 GR-.77200rnn.-02 61 .26785714, 07 c 5 ζ, ζ C RI .11500000. 5 7 ζ C 0 C 5 7 c c 01-0144435470 AXIAL STRAIN •115 SACC • .117500Cc. •115250cc• .1155npp •116000c •1165nnn. .11675cn. .117250C. .11775000 •1185506c• *118250nr* •119: none .11975000, .11575GGF; .11625crc. .11770000 .1185nnr. .11875nn. •119250CC+ •11950nn» •12000CGG• CASE NO.

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GR-•7720000002	OZ GTHET	ETA7720700002	02 6210290000,-01	01 RG .19300000.	100 • OI-
. 11. •63076036•	(7 (12	2 .217/4193, 07	C13 .95046082.	06 C22 •28064516•	116. 07 C44 .20000000 06
0114442547,	nk 421	.16166128, 06			
AXIAL STRAIN	9061	61099302			
œ.		SIGMA R	SIGMA THETA	SIGMA 2	צ
•115. CC75• 01	•	- 1	•	540	5736/20>02
11540000			0026600.	.19038570.	59233024,-02
.1155CC00 01		173300000 62	3497306.	8728440 • 0	
• 0000 /9 • 0000 /9		550000	.51440000 C3		1.664103412.107
-					
_			•	0	68495208,-02
1760000		•91100000 v1		0.0	-,70334793,-02
11820000			24925100, n4	0	72170241,-02
.118500000 D1		///000000 01 /4260000 02	3137880C+ 04 37391400- 04	-18233080, 05	- 74001632 - 02 - 7453 04 / 100
1940000	•	32/60000+ 02			
11960000	•			•	1947223002
2020000	•	\$62C000 •	•	•	612880900Z
2060000	•		•	7611920. 0	03100220,-02
12160066 01		10607000 03 12806000 03	67855200, 04	-17734730, 05	8490870002
12180000	•		79770700	7587080 0	
.1222n00r, n1	•	18042000, 03		7516570, 0	90312760,-02
260000		C875000.	538600.	0 • 0	92107210,-02
•12300000 01		23879000, 03	97368300, 04	17361970, 05	9389829002
œ		SIGMA R	SIGMA THETA	SIGMA Z	צ
2300000	•		4232, 0	5644291.	93898293,-02
1370000.	•	16081100, 03	8745656. 0	•	1058082301
.15100000, 01		10349302 • 03 	43013857, 0	_	1176109401
	•	001300339 UZ 26548513- 02	5319406. 0	156442919 04	124333734-01 14000533-01
19300000	·	14859259,-02	32664704.		-1526090701

C44 .200000000 06 NU3--16000000, 00 -.62277879.-02 -• 82899804 •-02 -.65385670 -- 02 --83521549.-02 --84764460 --02 **--860067**10 •-02 --86627580 -- 02 -- R7246340 -- 02 --87868910--02 --A8489370--02 --89109680 --02 -- 90349910 -- 02 --92829260--02 --94068400--02 --94687850--02 -- 14908340e-Ci -.17548641**--**01 --20161758--01 --84143085--02 --69729660.-02 -- 409649606----91589830+-02 --92209593•-02 --93448890--05 -- 9466 7860--02 --12225211-01 --22756542--01 C22 .28064516, 07 RG .12900000. 01 NU2 .900000000 00 AA2--500000000-01 NU .35000000 00 40 20 40 40 40 70 90 40 40 40 90 03 60 03 03 03 02 020 02 -.32438244. -.324382449 -.32438244. --32438244. .16761200, .16366300, .15556000. .15140HOO. .142°080°, 13856200 .13415100, 12968000 •12055200. .10158700. .91764000, .76616000, -.32438244. -.32438244 .14719000. .12514600 •11589900° .11118600, .10641600. .15964500. .96703000 .86767000. .81718000 62--58700000-02 C13 .95046082 • 06 AA1-.49000000.-02 NU1 .30000000. 00 .20900060, 07 04 S **0** 3 S 5 Ç 05 05 0.5 5 05 5 0 04 -.44334320. 05 SIGMA THETA .37683763, -06496799---46015500. -048900630. --45177970. -045597470. -.47260650, -.47672820, -06468083490. -.49307100. -.50518300, -.51318970, -.51717380. .26876439, .20651805. .14722067. .13038193, -.46432040. --468470HC+ -.48492780. -.49712200, -.50115920. -.50919300, -.52114440. -.52510210, .17153759, GTHE TA- . 18270000 -01 w C12 .21774193, 07 03 03 04 2 4 03 .14449382,-n7 RO .59000000 00 -20/000000-03 •200000000 06 .150000000.07 90 -.11898000, -.78135975, --23849000+ -.84299nn. --1n8796nn. --13346500. --14586200. --24645579. -.13838231, -.41155516, -.16838598. -.35844000. -04789C00. -.96528nng. -.15830000 -.18329700. --19585200, -.20844500° -.22108nno. -.24645400, -.59981000. -.72119000. -.12111100, --17077800. --23374900 A2--16166178. -- 610786701A-02 SIGMA R **62** F.2 C11 .63076036. 07 RI .56000000. 00 A1-.14443547, 06 F1 .45000000 07 .26785714, 07 GR- . 15270000 . - 01 CO C Ç C c C C: C C. C 0 Ċ 200 AXIAL STRAIN £60000000 .5615nnc. .5630000° .5660nnn. .575nnnn .578C0000. .57950CCO. .5840000° .5870000° 5885nnon. .59000000 .730C0006. .87000c0T8. .10100000. .1150000p •5645nnn. .5675nnn. .57n5nnn. .572: nncc. .57350nnc. .5765000C. 581000C+ .582500nn. .585500cc+ .5900000c •1290nno. . 569 COOP. CASE NO. 5

C44 .200000000 06 NU3--160000000, 00 -- 70994272,-02 -- 72068007 -- UZ -- 7314(551 -- 02 --74211935--02 --75282220•-02 -- 76351454 -- 02 --77419658•-02 --78486893•-02 --80618582 --02 --81683086 --02 --838097003-02 --85933280 --02 **--86984000 --**02 -- 88054090.-n2 -- R9113540 -- 02 -- 90172430,-02 --91230730•-02 -.20127086,-01 -- 79553187 -- 02 --82746780 -- 02 -- 44871830 -- 04 -.92288520.-02 --14808741 --01 --174872759-01 --92288540,-02 -.12069798.-01 --227405889-03 z o C22 •28064516• 07 O NUZ .900000000 00 AA2--500000000-01 GIHFTA--1827CCCC+-01 GZ--5870CCCC+-02 KG -1310CCCC+ NU .350000000 00 03 03 60 03 03 03 03 60 03 002 002 000 03 03 03 03 03 60 60 33 60 60 .23121124. .23121128. .23119321, SIGMA Z -.16003n00. -00087064--.63208000. -- 73592000 -.84710000. .23119321, .23121128. -.26466000 -636789000. -040674000. -.44172000. -.53590000. -.58301000. -.68307000 SIGMA Z -.18251000 -.20747000 -.23488000 --29642000. -.33123000. -.79062000. -.90534000. -.96530000 -.10269300, C13 .95046062, 06 AA1--49000000,-02 NUL .300000000. UN •20900000• 07 05 05 5 -.34973350. 05 5 05 05 05 5 .53639122. 04 70000 05 SIGMA THETA SIGMA THETA .30265707, .21540295. -.35716580, -.37188070, -.37916520. -.4698049C. .38718132, .19116068. -.36454800, -04967004--.4148R99G. --42190010. --43579280. --44267600. --45632010, .25019018. -.38640220 -.39359230. --40783540. -.42886770, -.44951850, -.4630822C. -.47648930. -.48313550. -048974440 W C12 .21774193, 07 40 03 3 C 40 40 2 04 -.24243101. 03 --31479012 -- 02 •30000000-01 .20000000 06 E2 .15000000 07 RO .61C00000. 00 H2--16166128. 06 -.15708030. -.59029810. -.31666006. -.21767900, -.34523084. -·196n2146, -.11149719, -.476k7nun. -06084469-- BO385000 --.182443CD, -·19999030 · --27157200+ -.28979900. -.34522800. -03016695*---11276509. -113070200---16504400+ -.23550800. --25347300, --30815300° --14/79600. --32663100, SIGMA & --61790623--02 SIGNA R 25 C11 .63076036. C7 A1--14443547. 06 RI .560000000 00 E1 .45000000 07 07 GR-.18270000.-01 c ζ C C ć C S C c c C. ç Ċ C. 01 25 .26785714. AXIAL STRAIN 56n' ann 5675000n · .7500ccor. 16300000 5625nnrn. .565000C. .57000007e .5725cco. 575 none .4775cnnc. 58250rno. •585conco .58750000 •59250000· .595'.nncn. •597500CC+ . 60000000 • 202000c *000u5209* .610000C+ .6100000n. •89000co+ .1175G00° .1310000. 59000nn .6C25000. œ CASF NO.

C44 .200001004 06 NU3--16000000, 00 70-017416616--- 26048165---61215752.-02 -.63357643,-02 -.68676925.-02 --13960636+-02 -• 79213638 •-Uz -->6200011-00 --65135023.-02 --669004024--02 -- (044]867--UZ --72203026.-02 -- 15/1402/0-02 70-1/02/04//---- any 25 60 y -- 02 --844403803-02 -- 62700799 -- 04 _ C22 .2d064516, 07 •13400000• OI NUZ .900000000 00 AA2-.530000000.-01 NU .35000000. 00 0 Š 40 30 90 5 40 70 -.23214200. --21636500. --21077500. -.22672700 --22214300. --21159503. -.21063500. -.21406300. -.227E3200. --21536400 -.21311700. -.21230000 -.21641800. --21934700 -.23139400. -.21115090. --22665300 <u>ა</u> GTHF.TA--18270000-01 GZ--58700000-02 C13 .55046082, 06 AA1--490000000-02 NUL .30000000. 00 E .209000000 07 C5 05 5 --22734/50, 05 Ç Ç S -.2401095--.26508210, -.31332800. -.25267010. -021734180. -06114605--930146910. -.3481423C. -.35950410. -.37074730, -.39290230. --41462360. -. 32505760. -.33666100. -- 3018810D. -·40381250. C12 .217/4193. 07 5 ç 10-400000 nde+ E2 .15000000 C7 RO .64000000 OF •20cconno • 06 a2-.16166128, 06 -.165/7000. -.51660700. -. 70155000. -88220000 --10884600. --14971-00 -.21476160. -.28388noG. -.33813000 · --12901900. --17091500. -.19260400. --23/3/000. --26041400. --30774900. -.33200900. SIGNA R --67744076--02 25 C11 .63076936, 07 71-11444747. PA RI .56000000 00 GR-+1827000C+-01 E1 .45000000 67 G1 .25785714, C7 Ç C AXIAL CTRAIN * 564. COOL •57650CDC• SAL JOUC . • 560. COOC. · SKROCKFC .597.nnn. •608000,00 .61200009 •61600000 •620:00nr .572crnn. · PRDC CPCC • .596. rrr.: • 0000 2509• •568CCOOC• • 0.0000.39• • 22000000 CASF MO.

--89644880,-02

-.24796900.

-.24196900.

-.25442700

-.23643809.

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-.42532890.

-.35664500.

. SZRJOODE.

.632 none •635CDDDD• *0000ccb9

-. 38164500,

-.40699100. --43267700,

--43593460, -.44644150, -.45685340.

--86177490.-02 --67912320,-02 -.91375410.-02

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.33221127; .25573613,

-.76475401, 03 -.31576628, 03 --28898765.-nz

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.1543635B. ·15436358 · .15436354. .15436358, .15436358. .15476358.

SIGNA THETA .68841549.

.50525161,

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-.43267969. -.24951580, --14339890.

•6400CCC0• •780000C+ •920000Ce •106ccnon• •1.200000+ *1340000°

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SIGMA R

.39913472. .28731247,

--17572949.-01 --20241925 -- 01 --I4854295 -- 0

--22876791 --0]

C44 .200000000 06 NU3--16000000, 00 --18352652,-01 --18413281,-01 - - 18716400•-01 -. 18777018,-01 -.18473910,-01 --18534533--01 --1859515/•-01 --18655779:-01 -·18837635,-01 -.18898253,-01 --18958869--01 --19019484 --01 --1308061----19140711.--01 --19201325,-01 --19261939•-01 --193225534-01 --19383165,-01 --19443780 -- 01 --19504393,-01 --19565007,-01 -.19565011,-01 --22260466 --01 --24922049 --01 --2755652,-01 --30176306,-01 --32779242 --01 ¥ 0 C22 •28064516• 07 NUZ .90000000 00 .18800000 AA2-.500000000.-01 .20900000 07 NU .35000000 00 02 02 02 020 90 40 70 -.63296501, -.63296501, -.63294694. -.63294694. -.63296501. .22207000 .21934900, .21661500. .21110800, .20833600. .20275300, .19712000, .19428500, ·18857930 · 18282500 .17410400, -.63296501, •21386800 · .20555000, .19994300. .19143800, •18570800 · .17993000 .17702200. .17117400, .16823200. .16527900. 2 GZ--5870C000.-02 C13 .95046082, 06 AA1--49000000-02 NUI .30000000. 00 05 05 9 04 400 05 90 05 05 05 05 -.5012c040, 05 S 9 05 05 .30454427, 04 SIGMA THETA SIGWA THETA -.50317950, -.50515540. .17213923, -.50712790. -092606940--.51106360. -.51498660, -.52279490. .26065894. .22878115, .18654643, -.513026RO. -.51694320. -.51889700. -.52084750. -.52473910. -.52668030. -.52861850, -.53055370, -.53248590. -.53441480. -.53634100, -.53826410, -.54018410. .20489926; GTHE TA-. 18270000 -- 01 u C12 .21774193, 07 03 6 10 -.32760367. 03 -.14407536. 03 -17545678 -- 02 E2 .150u0000 07 .200000000 06 RO .118C0000. 01 -.65801000. -.56642255. -.32795000. -.59183000. -.85700000 --13240537, --13093000+ -.19651000. --26219000. -.39380000. -.45972000 · --57574000 --72426000. -- 79059000 --92350900. -.99CC700G. --11234400. -.88519529· •00000000• -.65410000. --10567200. --11902400. -.12571100, --13240600. H2--16166178. SIGMA R -- 61R85562,-02 **62** C11 .63076036, 07 81-.14442547, 06 •45000000• 01 .26785714, 07 GR-.1827CC00.-C1 .1150ccon. c1 5 5 5 5 5 C 5 0 5 10 0 5 0 0 5 5 5 AXIAL STRAIN •116950n0• .11725000. .1180noon. .11560000. .11590000. .116:5nn. .1162nnrr. .11710000. •11770con• -118C0000. .14600000 .174cn000. .115. nnon. .11515000. .11530000 .11545000. .115750CC+ 6350CC . .1165nnnr. .11665000. .1160000C. •1174CC0C+ .1175500n, .11785nn. .132C0000. .1600000n. .18800000 œ œ CASF NO. ច

CASE NO.

C44 .200000000 06 NU3--160000000 CO --27053064.-01 --17346700--01 --24374609--01 -.29703207,-01 --16830171•-01 --17449933--01 --18171901----18275031,-01 --18378085 •-01 --18584146--01 --18790147--01 -.18893129,-01 --21658969.-01 --32331298+-01 -.16933529.-0 --17036859.-0 --17140165 --0 --17243445.-0 --17553141 --0 --17656330,-0 --11759494.-C --17862639.-0 --17965765-0 -- 18068872 •-0] --18481122·-0 --18687154,-0] -.18893127.-C 0 C22 .28064516, 07 NU2 .90000000 00 AA2-.50000000.-01 KG .19000000. NU .35000000 00 03 22222 .52647000. •32105cno .21273000 -.16881678. -.16879872. -.16881678. .65471000. .59149000 .49331000. .4597c000. .35634000. .13854000. -.16879872, -.16681678. .62333000 •55921c00• .42567000. .24924000. .10085000. SIGMA Z .39121000. .28535000 .17563000 •24340000 · SIGMA 2 .62790000 -.14460000 -.53670000 62--58700000--02 C13 .95046062, 06 NU1 .30000000, 00 AA1-.490000000-02 .20900000 07 05 9 3 S 500 05 9 50 05 05 50 000 400 3333 05 5 S 02 3 SIGMA THETA SIGWA THETA .45835118, --43911950. .31048012. .26139628. -.50239440. •39346308 · .28301599. -044253 700. -04456440 -044934170 --45272910, -.45610650, -.45947420. -.46283200. -.46618040. -.46951890. -.472848nC. -.47616770. -.47947610. --48277920. -.46607100. -048935370. -.49262720, -.49589190. -.49914760. -.50563230, .34610198, GTHETA--18270000.-01 u .217/4193. 07 40000 60 53 03 03 6 č 6 60 40 40 04 40 40 40 4 40 4 4 --16513580,-02 E2 .15000000 07 •200000000 06 --10000000--01 •120c0000• 01 B2-.16166128, 06 -.21620389. -.95630000 -.19695561. -.13206695; -.84/05852. --49084537 -.38444000· -.46133000. -.57854nn. -.15666500. -.16669900. --1767590n, -19695500. -.19158000· --28/85nuc. -.676n5n0n -.77486nn. -.87196nnn. -.97036000. -10690400 -.11680100. -- 126/2700. -.13667800. -.14665800. -.18684400. SIGNA R SIGMA R --64158212,-02 C12 **9** 62 C11 .63076036. n7 .450000000 07 .26785714, 07 GR-. 18270000,-01 A1-.14447547, 06 •11500000 01 -2010 910 5 5 5 0 AXIAL STRAIN .116250CC. .11825con. .13400000. .17600000 11575005. .117700n-.11775cnn. .11850000. .119(nnon. .14800000 .16200000. 11525000 .1155ngon. 11600000 1650000 .11675rns .11775cnc. 750000 •1180000° .1187500C. .1192500C. 11950000 .11975nnc. .12000000 • 190cnn00 • •115: DOOC. 1200000 œ œ E ទ

CASE NO. 12

F1 .45000000. 07 F2 .15000000. 07 NUI .30000000. 00 NUZ .90000000. 00 NU3-.16000000. 00 GR-:5280000--02 GTHETA--52800000-02 GZ--71400000--02 RG -12900000 01-G2 .2GCOCOCO. 06 AA1-.49000000.-02 AA2-.50000000-01. RO .59000000 00 F .30000000 06 NU .35000000 00 RI .56000000. CC G1 .26785714, 07 CASE NO.

C11 .63076036. n7 C12 .21774193. 07 C13 .95046082. 06 C22 .28064516. 07 C44 .20000000. 06 HI-.1444754/, Pt. 92-,16156128, 06

AXIAL STRAIN --64652585.-02

\$1138 K • \$6150000 • \$6450000 • \$6450000 • \$6600000 • \$6600000 • \$6600000 • \$670000 • \$730000 • \$730000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7300000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7350000 • \$7300000 • \$7300000 • \$7300000 • \$7300000 • \$7300000 • \$7300000 • \$7300000 • \$7300000 • \$7300000 • \$7300000 • \$7450000	-01 -68269400 04 02 -62728300 04 02 -57217400 04 02 -54286400 04 02 -462865200 04 02 -35473200 04	v • • •	x .
	.672178300 .57217400 .51737000 .46286460 .45865200 .35473200	.81339700; .80315400; .79297300;	
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	26.776600	73535100-05	20-02061620-
	19465 100	- 72626300+ 04	. 67001676
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	• 3718nnng.	70032100, 04	29412048
	•		30140403 02
	0366476000, 03	68385700. 04	3086 7438 02
	-11790600	67586900. 04	3159321302
	0316906903, 04	06803800. 04	3231 7666 02
	0221997400, 04	•	•
	0227062500, 04	65284800. 04	3376291802
c c	0232102000, 04	64548500, 04	
	7237116300. 04	•	
	SIGMA THETA	SIGMA 2	ж Э
	113903	•18862908 • 03	3520527702
or .41627825.	81237422.		424835019-02
Fin .2361762	. 7	.18862908, 03	49918092 02
•	51849422.		•
			65024866 02
01 44444444	39409622.	_	72645478 02

C44 .200000000 06 NU3--160000000 00 --15459059 --02 --16723339.-02 --17983129,-02 --19238519.-02 --20482229--02 --21736478,-02 --22979210--02 --24417918 -- 04 --25452668 --02 -.26683518.-02 -.27910558 ·- 02 -.29133888.-02 --32782291.-02 --3399146/--02 --35197307.-02 --36399837.-02 --37599167.-02 --38795336 -- 02 --30353577,-02 --31569677,-02 -.39988426 -- 02 z o C22 •28064516• 07 KG •13100000 01 NUZ .90000000 00 AA2--500000000-01 NU .35000000 00 400 40 40 40 90 40 04 90 9 04 40 -.75223900, -00990969-SIGMA Z -- /3206600. -- 11424500. -.67841500. --66128400. -.64466100. -.61289900. -.59773800. -.56881100, -.55502803. -.54168300. -.52877000. -.51627900, --49253400. --48126300+ -.45988500. --62653600. -.58304600, -- 50420400 -.47038300. GTHETA-.52800000.-02 GZ-.71400000.-02 C12 .217/4193. 07 C13 .95046082. 06 NUL .30000000. 00 AA1-.49000000.-02 E .300000000. 36 400 35 90 40 6 40 *1083114C, 05 40 č 04 40 60 Č 04 40 40 SIGMA THETA .98855200. .52863500. -.49637000. .89487109. . H0205400. .26244600. .17525900. .882000C. .31140000. -.61867000° -.16614100, --24971300. -.41481700. -.57727100. -.65753200. . /1008 700, .61895200. .43912100. .35039400. -.33260300. 03 03 53 6 6 č Ç Ę. 03 03 03 03 6.0 č 20 ·00009094 10-100000006 F2 .15000000 07 •200c0000 06 RO .61000000 00 A2-.161c612H, 06 .8752Cnn. .12450900. .15/13con. .20959000. .22963000 .27127ngn. .26647000. .23914000. .20290000. .18546000. .24564nno. .25774000. .27047n00. .26212000. .25234000. .22265000, .17997000. 15393000. .26599nn. SIGNA 9 -- ¢18653¢6.-02 25 C11 .63076036, 07 81-.14443547, 06 F1 .45000000 07 RI .56000000 PT GR-.52800000 -02 G1 .26785714, n7 c C AXIAL STRAIN •5625cccc. **.** JUUULU95 •5675nnn. .5700 0000 .57250000. .575.0000° .5775c000. •5870000• .5825000c . 585. rrr. .58750000 .590000000 .5925cnoo. .595cncoc. •5975000°• • 200000079• .6025000r. •6050000n• .60750000 .6100000. . 565. COOP. CASE NO.

--46921291,-02

-.11152739. -.96020392. -.65213814.

-.13491556.

.15389312. 03 .87360029. 02 .49701699. 02 .26313451. 02 .10806525. 02

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.6100000 •75000000. •8900000e .10300000 .11700000 13100000

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-.17259390,

-- 76804799 -- 02

--39988434 -- 02 -.54170916,-02 -.61608139.-02 --69165619.-02

.25621357. .25621357. .25621354. .25621357. .25621357.

-.23910698, 03

SIGMA THETA

SIGMA R

SIGMA Z

.25621357.

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NU3--16000000, 00 RG -13400000, 01 NUZ .90000000, 00 AA2-.500000000.-0 NI .3500000 00 62--71400000-02 NUL . 300000000: 111N AA1-.49000000-02 F . שחרוחרוחר ה GTHFTA-.52800000.-02 Ċ 9 F2 .15000000 67 • 20000000 RO .64PPPPPP 2 Ç F1 .45000000 07 61 .26785714. 07 GR-. 57800000 -- C2 *KANONNO. CASE NO.

C44 .20000000 06 C22 .28064516, 07 C13 .95046082, 06 C12 .21774193, 07 Cil .53075036. 07

nl-.14443547, 06 82-.16166128, 06

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AXIAL STRAIN

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APPENDIX F

Nomenclature for Computer

Print-Out Sheets

APPENDIX F

Nomenclature for Computer Print-Out Sheets

1. Pyrolytic Graphite Properties

E1 - Young's modulus, a-direction, for pyrolytic graphite, psi
E2 - Young's modulus, c-direction, for pyrolytic graphite, psi
NU1 - Poisson's ratio, c-a direction, for pyrolytic graphite
NU2 - Poisson's ratio, a-c direction, for pyrolytic graphite
NU3 - Poisson's ratio, a-a direction, for pyrolytic graphite
G1 - Shear modulus in c-direction in pyrolytic graphite, psi
G2 - Shear modulus in a-direction pyrolytic graphite, psi
AA1 - Total thermal expansion in a-direction in pyrolytic graphite, inch/inch
AA2 - Total thermal expansion in c-direction in pyrolytic graphite, inch/inch
C11
C12
C13
C22
C44
B1
B2

2. Substrate Properties

E - Young's modulus for substrate, psi

NU - Poisson's ratio for substrate

GR - Total thermal expansion in radial direction for substrate, inch/inch G THETA - Total thermal expansion in hoop direction for substrate, inch/inch GZ - Total thermal expansion in axial direction for substrate, inch/inch

3. Dimensions

RI - Radius at free surface of pyrolytic graphite coating, inch

NO - Radius at coating - substrate interface, inch

RG - Radius at free surface of substrate, inch

4. Output Data

R - Local radius, inch SIGMA R - Radial stress, psi SIGMA THETA - Hoop stress, psi SIGMA Z - Axial stress, psi UR - Radial displacement, inch AFRPL-TR-65-57

ADDENDUM

TEST OF THE RESTART CAPABILITY OF A PYROLYTIC GRAPHITE COATED NOZZLE

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AFRPL-TR-65-57

INTRODUCTION

During the program described in the main body of this report, five subscale and four fullscale pyrolytic graphite coated nozzles were motor tested with an advanced high performance propellant. These firing tests demonstrated that nozzles of useful size coated with pyrolytic graphite can provide good performance under severe service conditions. Although feasibility was clearly demonstrated for pyrolytic graphite coated nozzles, further work remains to be carried out in several areas to fully exploit the capabilities of this unique, lightweight nozzle design. Optimization of the coating-substrate composite for maximum erosion resistance is needed. One of the facets of particular interest is an investigation of the restart capability of pyrolytic graphite coatings.

Restart capability is of such current interest that Atlantic Research Corporation requested the permission of the Air Force to include an additional firing test in the current program in which a subscale nozzle would be re-fired in a second test. Permission was granted for this restart test and this addendum documents the successful completion of this additional task.

RESULTS

The pyrolytic graphite coated insert selected for the restart test was the insert first tested in firing EPb-6. In this first test an erosion rate of 0.64 mil/sec was measured for a firing duration of 37.3 seconds at an average motor pressure of 530 psi. The insert appeared to be in good shape following the first test and microscopic examination revealed no delamination flaws before or after firing EPb-6. The coating on this insert was the first prepared on the fibrous graphite type of substrate. Careful macroscopic examination of the surface of the once-tested coating indicated areas of surface roughness related no doubt to the fact that the coating quality had not been optimized for this system. A close-up photograph of the coated insert after firing EPb-6 and prior to the restart test (firing EPb-10) is shown in Figure 34. Nevertheless, it was concluded that this insert was suitable for studying the restart behavior of a pyrolytic graphite coating.(C)

The once-tested insert was assembled into the standard subscale nozzle unit (see Figure 15). The ATJ graphite and edge-oriented pyrolytic graphite entrance pieces were also re-used from firing EPb-6. Thus, the entire nozzle contour from an upstream area ratio of about six through the throat region was made up of components which were undergoing restart operation. Several of the back-up and insulation components were replaced only because of mechanical damage during disassembly after firing EPb-6 and before plans were made for the restart test. Overall, then, the restart test was a good test of all the critical components of a pyrolytic graphite coated nozzle. (U)

The restart test was firing EPb-10 and consisted of a 15.1-second duration test at an average motor pressure of 532 psia with 6550°F propellant (APG-112). The firing was a complete success with an average erosion rate based on before and after test measurements of the throat diameter of 0.53 mil/sec. The throat was somewhat oval after the first firing with a difference in the maximum and minimum diameters of some 20 mil. After the second firing the ovalness was still the same which indicates no further selective erosion. The motor pressure-time trace for the restart firing EPb-10 is shown in Figure 35. A close-up oblique view of the nozzle throat region looking from the entrance end is shown in Figure 36. (C)

Microscopic examination of the polished cross section of the coating after its second test revealed no change in structure and no delamination flaws. Thus, no degradation of the coating quality was visible after either one or two firing cycles. The remaining coating thickness at the edge of the coated section, where it was thinnest initially, appears insufficient to allow further testing, but the cumulative firing time on this insert is already 52.4 seconds. (U)

The erosion rate observed in the second firing was somewhat lower than in the first test. However, if an induction period of only about two seconds prior to the onset of erosion were assumed, the erosion data would be comparable. It is indicated, therefore, that the rapid heating of the coating surface producted by thermal analysis is consistent with the experimental data. (U)

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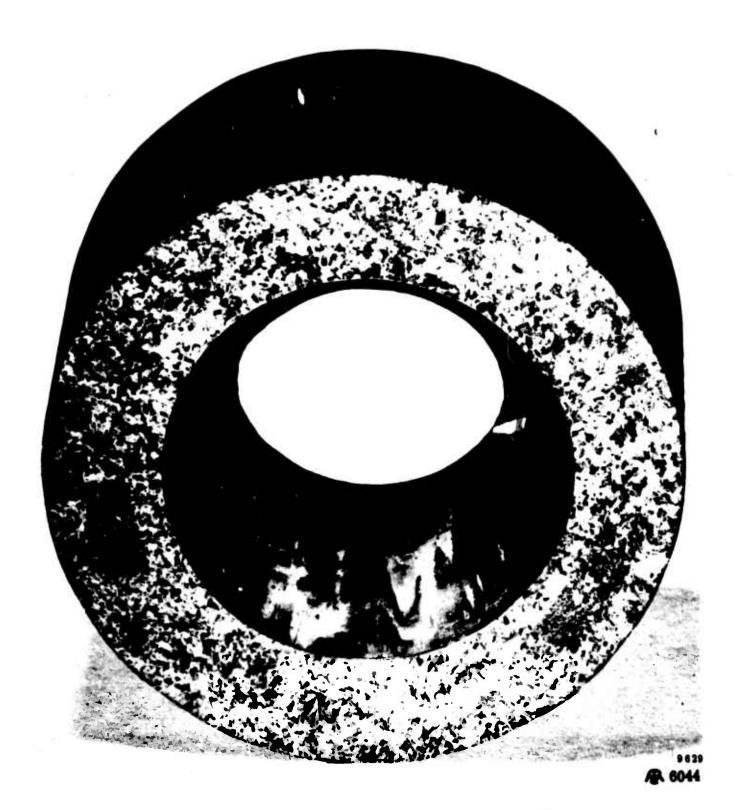


Figure 34. Macro-View (3X) of Coated Insert Prior to Restart Test.

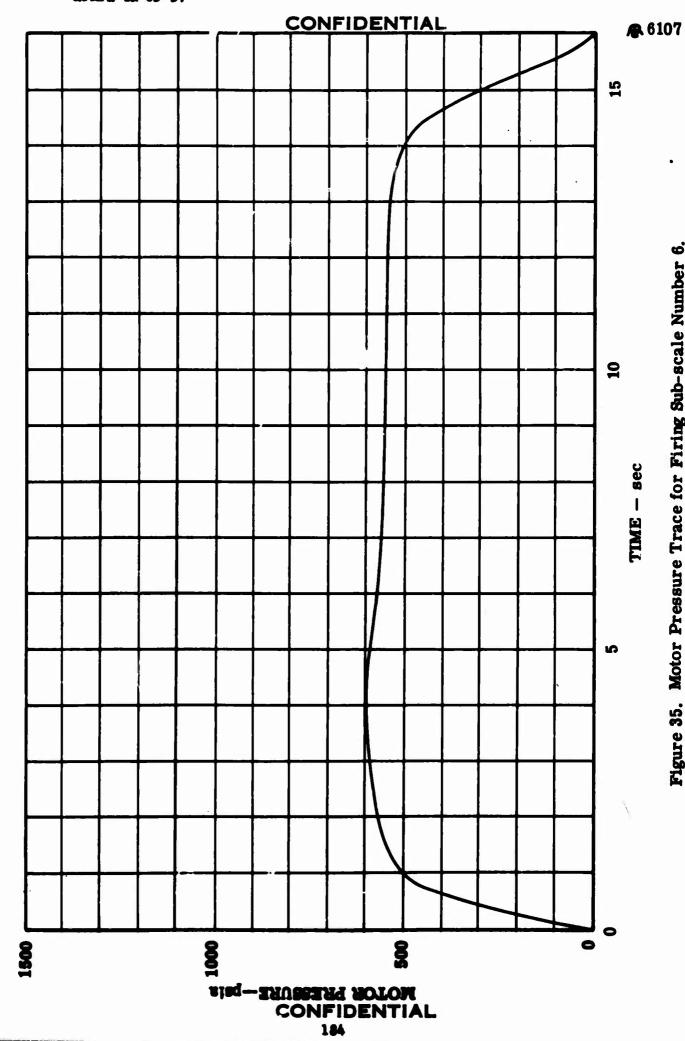


Figure 35. Motor Pressure Trace for Firing Sub-scale Number 6.



Figure 36. Close-Up Oblique View of Nozzle (from Entrance) after Restart Test.

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CONCLUSIONS

The results of the work described in the main body of this report demonstrated that pyrolytic graphite coatings which are free of delamination flaws can provide good nozzle performance. A second firing test of one coated insert described in this addendum demonstrated that such a nozzle can withstand restart service conditions. The complete criteria for suitable restart performance and the duty cycle limitations could not be defined by a single test, but the successful performance of the insert in two firings proves that pyrolytic graphite coated nozzles are good candidates for restart operation.

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Pyrolytic graphite coatings have been found to be highly erosion resistant under severe rocket nozzle conditions. The objective of this program was to demonstrate the feasibility of such coatings for nozzles up to 2.3-inch diameter in firings at 700 psi with a 6550°F propellant. This objective was accomplished.

Stress analyses and thermal analyses were carried out in support of the design, fabrication, and motor testing of nozzles of both 1.1-inch and 2.3-inch diameter. Thermal analysis indicated the potential of pyrolytic graphite coatings for lightweight nozzle designs. The results of the stress analyses for coated composites were correlated with experimental evidence of delamination cracking in the coatings. Critical stress levels were identified for both radial tension in the coating and axial tension in the substrate. The deposition process was improved to produce crack-free coatings 50-mil-thick on conventional graphite substrates and 100-mil thick on a fabrous graphite substrate.

Nine motor firing tests were made and good performance was demonstrated with both subscale and full-scale nozzles. The presence of microscopically observed delamination cracks was found to destroy the integrity of pyrolytic graphite coatings during nozzle service. For coatings without major cracks coating integrity was achieved, however, and erosion rates of acceptable magnitude were measured for subscale nozzles for durations up to about 40 seconds. In full-scale nozzles satisfactory erosion rates were measured for fixings of durations up to 60 seconds. Optimization of the coating to provide minimum erosion rates should result in still further performance improvements.

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Security Classification

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